



BTX III

X-Ray Diffraction Analyzer

User's Manual

10-015490-01EN — Rev. 3
June 2021

This instruction manual contains essential information on how to use this Olympus product safely and effectively. Before using this product, thoroughly review this instruction manual. Use the product as instructed. Keep this instruction manual in a safe, accessible location.

Olympus Scientific Solutions Americas, 48 Woerd Avenue, Waltham, MA 02453, USA

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This document was prepared with particular attention to usage to ensure the accuracy of the information contained therein, and corresponds to the version of the product manufactured prior to the date appearing on the title page. There could, however, be some differences between the manual and the product if the product was modified thereafter.

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

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List of Abbreviations

| | |
|---------|--|
| ALARA | as low as reasonably achievable |
| AMCSD | American Mineralogist Crystal Structure Database |
| BAG | Bundesamt für Gesundheit (Schweiz) |
| CCD | charge-coupled device |
| DHCP | dynamic host configuration protocol |
| EFUP | environment-friendly use period |
| FWHM | full width at half maximum |
| HVPS | high-voltage power supply |
| LAN | local area network |
| OSS | open source software |
| PXRD | powder X-ray diffraction |
| RIR | reference intensity ratio |
| RöV | Röntgenverordnung (Deutschland) |
| StrSchG | Strahlenschutzgesetz (Österreich) |
| SUVA | Schweizerische Unfallversicherungsanstalt |
| WLAN | wireless local area network |
| XRD | X-ray diffraction |

Labels and Symbols

Safety-related labels and symbols are attached to the BTX III X-ray diffraction analyzer (see Figure i-1 on page 2). If any of the labels or symbols are missing or illegible, please contact Olympus.



X-RAY ON



PRODUCTION de RAYONS X



Figure i-1 X-ray warning labels

NOTE

The radiation symbol may differ according to your location (see “Safety Symbols” on page 7).

The rating label is attached to the BTX III analyzer. The label symbols are described in Table 1 on page 3.

Table 1 Content of the rating label









| Item | Description |
|--|--|
|  | <p>This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions: (1) this device may not cause harmful interference, and (2) this device must accept any interference received, including interference that may cause undesired operation.</p> |
|  | <p>The CE marking is a declaration that this product conforms to all the applicable directives of the European Community. See the <i>Declaration of Conformity</i> for details. Contact your Olympus representative for more information.</p> |
|   | <p>The WEEE symbol indicates that the product must not be disposed of as unsorted municipal waste, but should be collected separately.</p> |
|  | <p>The regulatory compliance mark (RCM) label indicates that the product complies with all applicable standards, and has been registered with the Australian Communications and Media Authority (ACMA) for placement on the Australian market.</p> |

Table 1 Content of the rating label (continued)

| | |
|---|--|
|  | <p>The KCC marking is a declaration that this product conforms to all the applicable standards of South Korea. Contact your Olympus representative for more information. The MSIP code for the BTX III is the following: R-R-OYN-BTXIII.</p> |
|  | <p>The China RoHS mark indicates the product's Environment-Friendly Use Period (EFUP). The EFUP is defined as the number of years for which listed controlled substances will not leak or chemically deteriorate while in the product. The EFUP for the BTX III has been determined to be 15 years. Note: The Environment-Friendly Use Period (EFUP) is not meant to be interpreted as the period assuring functionality and product performance.</p> |
|  | <p>The direct current symbol.</p> |
| <p>SERIAL NUMBER</p> | <p>The serial number is in the following format: BTX-XXX where: XXX represents a three-digit number</p> |

Important Information — Please Read Before Use

Intended Use

The BTX III X-ray diffraction analyzer is designed primarily for analyzing a variety of powder sample types.



WARNING

Do not use the BTX III analyzer for any purpose other than its intended use. It must never be used to inspect or examine human or animal body parts.

Instruction Manual

This instruction manual contains essential information on how to use this Olympus product safely and effectively. Before using this product, thoroughly review this instruction manual. Use the product as instructed.

Keep this instruction manual in a safe, accessible location.

IMPORTANT

Some of the details of components and/or software images in this manual may differ from your instrument's components or software display. However, the principles remain the same.

Instrument Compatibility

The BTX III XRD analyzer is primarily a self-contained unit. However, it does have a series of I/O ports that can be used to connect compatible peripherals and connect it to a PC. The unit derives its required DC input power from the BTX III analyzer's AC adaptor.



CAUTION

Always use equipment and accessories that meet Olympus specifications. Using incompatible equipment could cause equipment malfunction and/or damage, or human injury.

Repair and Modification

The BTX III XRD analyzer does not contain any user-serviceable parts, apart from one exception: the measurement window. If the measurement window is damaged, the window assembly should be replaced as soon as possible. This User's Manual contains more details on this procedure.



CAUTION

In order to prevent human injury and/or equipment damage, do not disassemble, modify, or attempt to repair the instrument.

Safety Symbols

The following safety symbols might appear on the instrument and in the instruction manual:



General warning symbol

This symbol is used to alert the user to potential hazards. All safety messages that follow this symbol shall be obeyed to avoid possible harm or material damage.



Radiation warning symbol (International)



Radiation warning symbol (Canada)



Radiation warning symbol (China)

These symbols are used to alert the user to the presence of potentially harmful ionizing radiation generated within the XRD analyzer. All safety messages that follow this symbol shall be obeyed to avoid possible harm.



Shock hazard caution symbol

This symbol is used to alert the user to potential electric shock hazards. All safety messages that follow this symbol shall be obeyed to avoid possible harm.

Safety Signal Words

The following safety signal words might appear in the documentation of the instrument:



DANGER

The DANGER signal word indicates an imminently hazardous situation. It calls attention to a procedure, practice, or the like that if not correctly performed or adhered to will result in death or serious personal injury. Do not proceed beyond a DANGER signal word until the indicated conditions are fully understood and met.



WARNING

The **WARNING** signal word indicates a potentially hazardous situation. It calls attention to a procedure, practice, or the like that if not correctly performed or adhered to could result in death or serious personal injury. Do not proceed beyond a **WARNING** signal word until the indicated conditions are fully understood and met.



CAUTION

The **CAUTION** signal word indicates a potentially hazardous situation. It calls attention to a procedure, practice, or the like that if not correctly performed or adhered to may result in minor or moderate personal injury, material damage, particularly to the product, destruction of part or all of the product, or loss of data. Do not proceed beyond a **CAUTION** signal word until the indicated conditions are fully understood and met.

Note Signal Words

The following note signal words could appear in the documentation of the instrument:

IMPORTANT

The **IMPORTANT** signal word calls attention to a note that provides information that is important or essential to the completion of a task.

NOTE

The **NOTE** signal word calls attention to an operating procedure, practice, or the like, that requires special attention. A note also denotes related parenthetical information that is useful, but not imperative.

TIP

The **TIP** signal word calls attention to a type of note that helps you apply the techniques and procedures described in the manual to your specific needs, or that provides hints on how to effectively use the capabilities of the product.

Safety

Before turning on the instrument, verify that the correct safety precautions have been taken (see the following warnings). In addition, note the external markings on the instrument, which are described under “Safety Symbols” on page 7.

Warnings



WARNING

General Warnings

- Carefully read the instructions contained in this instruction manual prior to turning on the instrument.
- Keep this instruction manual in a safe place for further reference.
- Follow the installation and operation procedures.
- It is imperative to respect the safety warnings on the instrument and in this instruction manual.
- If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment could be impaired.
- Do not install substitute parts or perform any unauthorized modification to the instrument.
- Service instructions, when applicable, are for trained service personnel. To avoid the risk of electric shock, do not perform any work on the instrument unless qualified to do so. For any problem or question regarding this instrument, contact Olympus or an authorized Olympus representative.
- Do not touch the connectors directly by hand. Otherwise, a malfunction or electric shock may result.
- Do not allow metallic or foreign objects to enter the device through connectors or any other openings. Otherwise, a malfunction or electric shock may result.
- Make sure no BTX III components (screws, straps, etc.) have come loose or are lost in critical equipment being inspected. Thoroughly check your inspection area before and after an inspection to prevent foreign-object debris (FOD) that could potentially cause equipment damage, injuries, or loss of life.



Radiation Safety Warning

Do not open the system, disassemble, or modify any internal components. These actions could result in serious damage to the system and a health hazard to the operator.



WARNING



Electrical Warnings

- Before operating this instrument using mains electricity, you must connect the protective earth terminal of the instrument to the protective conductor (mains) of the power cord. The mains plug shall only be inserted into a socket outlet provided with a protective earth contact. Never negate the protective action by using an extension cord (power cable) without a protective conductor (grounding).
- Only use fuses with the required rated current, voltage, and specified type (normal-blow, slow-blow, quick-acting, etc.). Do not use repaired fuses or short-circuited fuse holders, doing so could cause electric shock or create a fire hazard.
- If there is any possibility that the ground protection could be impaired, you must make the instrument inoperative and secure it against any unintended operation.
- The instrument must only be connected to a power source corresponding to the type indicated on the rating label.



CAUTION

If an unauthorized power supply cord is used to power the instrument, Olympus cannot guarantee the electrical safety of the equipment.

**WARNING****High Voltage**

The BTX III XRD analyzer uses a 30 kV high-voltage power supply (HVPS) for X-ray generation. The permanent connection between the HVPS and the X-ray tube is sealed and shielded in such a way that no high voltage connector could accidentally get loose or disconnected inside the system. There is no high voltage risk to the user when using the BTX III analyzer under normal conditions. Should you notice substantial damage to the outside of the system, or suspect any internal damage after excessive shock, DO NOT turn on the system, return it to the factory for full inspection and potential repairs.

**CAUTION**

- X-ray tubes and detectors in this instrument contain beryllium metal in the form of coated foil. In its as-supplied state, the beryllium poses no harm to the user. However, if a detector or tube is damaged, contact with small particles is possible if the instrument is breached (for example, a window is broken or during window replacement). Intact skin is sufficient protection against this situation and washing with soap and water will effectively remove any beryllium contamination. If granulated beryllium embeds in an open wound, seek medical attention.
- Instruments with a damaged detector or tube must be returned to your local distributor or the manufacturer. Care should be taken to limit the release of beryllium from the instrument.

Warning Labels

Warning labels and pictograms could appear on the inspection system to alert operators about hazardous areas. Keep clear of such areas to avoid injury.

Richtlinien für den Strahlenschutz im deutschsprachigen Raum

Deutschland

Der Betrieb eines tragbaren Röntgenfluoreszenzanalysators ist in jedem Fall genehmigungspflichtig (§ 3 RöV).

Sorgen Sie dafür, dass in Ihrem Betrieb mindestens ein Strahlenschutzbeauftragter mit Fachkunde R2 nach der deutschen Röntgenverordnung verfügbar ist. Darüber hinaus sollten Sie die Bediener regelmäßig schulen lassen. Eine jährliche Unterweisung der Bediener ist vom Strahlenschutzbeauftragten durchzuführen.

Es ist i. d. R. ein Betriebsbuch (Nachweis der Betriebszeiten, Wartungsarbeiten und Störfälle) zu führen. Eine betriebliche Strahlenschutzanweisung ist zu erstellen und mit der deutschsprachigen Bedienungsanleitung den Gerätebedienern jederzeit zugänglich zu machen.

Das Strahlenschutztechnische Gutachten für das jeweilige Instrument wird bei Auslieferung des Gerätes von einem behördlich zugelassenen Sachverständigen erstellt und sollte jederzeit einsehbar sein. Spätestens nach 5 Jahren muss dieses Gutachten erneuert werden.

Die Genehmigungsbehörde kann weitere Maßnahmen zur Arbeitssicherheit festlegen.

Österreich

Der Betrieb eines tragbaren Röntgenfluoreszenzanalysators ist genehmigungspflichtig.

Es ist ein Strahlenschutzbeauftragter und die entsprechende Anzahl von weiteren, mit der Wahrnehmung des Strahlenschutzes betrauten Personen, zu nominieren. Der Strahlenschutzbeauftragte ist der Behörde bekannt zu geben. Der Strahlenschutzbeauftragte und die weiteren Personen haben eine entsprechende Ausbildung gemäß der allgemeinen österreichischen Strahlenschutzverordnung nachzuweisen.

Das Bedienpersonal (sofern nicht selber strahlenschutzbeauftragt) ist gemäß § 16 Allgemeine Strahlenschutzverordnung mindestens einmal jährlich vom Strahlenschutzbeauftragten gemäß § 29 StrSchG zu unterweisen. Dies muss dokumentiert werden.

Eine deutschsprachige Bedienungsanleitung sowie Handlungs- und Arbeitsanweisungen sind dem Bedienpersonal jederzeit zur Verfügung zu stellen.

Es wird empfohlen jeden Bediener mit einem amtlichen Dosimeter auszurüsten.

Schweiz

Der Betrieb von Röntgenfluoreszenzanalysatoren ist der BAG anzuzeigen (Bewilligungsverfahren).

Für den Einsatz von tragbaren Röntgengeräten ist für jeden Betrieb ein für den Strahlenschutz verantwortlicher Mitarbeiter zu benennen, der eine dem Schweizer Strahlenschutzgesetz genügende Ausbildung bei der SUVA (eintägiger Kursus) erfolgreich absolviert hat.

Die Bedienungsanleitung sowie weitere eventuell von den Behörden geforderte Unterlagen wie Arbeitsanweisungen und Anweisung im Falle von Störungen müssen den Bedienern zugänglich gemacht werden.

Tragbare RFA-Geräte mit offenem Strahlengang müssen im Zwei-Hand-Modus bedient werden.

Equipment Disposal

Before disposing of the BTX III, check your local laws, rules, and regulations, and follow them accordingly.

CE (European Community)



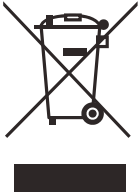
This device complies with the requirements of directive 2014/30/EU concerning electromagnetic compatibility, directive 2014/35/EU concerning low voltage, and directive 2015/863 which amends 2011/65/EU concerning restriction of hazardous substances (RoHS). The CE marking indicates compliance with the above directives.

UKCA (United Kingdom)



This device complies with the requirements of the Electromagnetic Compatibility Regulations 2016, the Electrical Equipment (Safety) Regulations 2016, and the Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment Regulations 2012. The UKCA marking indicates compliance with the above regulations.

WEEE Directive



In accordance with European Directive 2012/19/EU on Waste Electrical and Electronic Equipment (WEEE), this symbol indicates that the product must not be disposed of as unsorted municipal waste, but should be collected separately. Refer to your local Olympus distributor for return and/or collection systems available in your country.

China RoHS

China RoHS is the term used by industry generally to describe legislation implemented by the Ministry of Information Industry (MII) in the People's Republic of China for the control of pollution by electronic information products (EIP).



The China RoHS mark indicates the product's Environment-Friendly Use Period (EFUP). The EFUP is defined as the number of years for which listed controlled substances will not leak or chemically deteriorate while in the product. The EFUP for the BTX III has been determined to be 15 years.

Note: The Environment-Friendly Use Period (EFUP) is not meant to be interpreted as the period assuring functionality and product performance.

“中国 RoHS”是一个工业术语，一般用于描述中华人民共和国信息工业部（MII）针对控制电子信息产品（EIP）的污染所实行的法令。



电气电子产品
有害物质
限制使用标识

中国 RoHS 标识是根据“电器电子产品有害物质限制使用管理办法”以及“电子电气产品有害物质限制使用标识要求”的规定，适用于在中国销售的电气电子产品上的电气电子产品有害物质限制使用标识。

注意：电气电子产品有害物质限制使用标识内的数字为在正常的使用条件下有害物质不会泄漏的年限，不是保证产品功能性的年限。

产品中有害物质的名称及含量

| 部件名称 | | 有害物质 | | | | | |
|------|------|----------------|----------------|----------------|----------------------|---------------|-----------------|
| | | 铅及其化合物 (Pb) | 汞及其化合物 (Hg) | 镉及其化合物 (Cd) | 六价铬及其化合物 (Cr(VI)) | 多溴联苯 (PBB) | 多溴二苯醚 (PBDE) |
| 主体 | 机构部件 | × | ○ | ○ | ○ | ○ | ○ |
| | 光学部件 | × | ○ | ○ | ○ | ○ | ○ |
| | 电气部件 | × | ○ | ○ | ○ | ○ | ○ |
| 附件 | | × | ○ | ○ | ○ | ○ | ○ |

本表格依据 SJ/T 11364 的规定编制。

○：表示该有害物质在该部件所有均质材料中的含量均在 GB/T26572 规定的限量要求以下。

×：表示该有害物质至少在该部件的某一均质材料中的含量超出 GB/T26572 规定的限量要求。

Korea Communications Commission (KCC)



이 기기는 업무용 환경에서 사용할 목적으로 적합성평가를 받은 기기로서 가정용 환경에서 사용하는 경우 전파간섭의 우려가 있습니다.

KC (South Korea Community)

This device complies with the requirements of KN 61000-6-2 and KN 61000-6-4 concerning electromagnetic compatibility. The KC marking indicates compliance with the above standards.

EMC Directive Compliance

This equipment generates and uses radio-frequency energy and, if not installed and used properly (that is, in strict accordance with the manufacturer's instructions), may cause interference. The BTX III has been tested and found to comply with the limits for an industrial device in accordance with the specifications of the EMC directive 2014/30/EU.

FCC (USA) Compliance

| |
|-------------|
| NOTE |
|-------------|

This product has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the product is operated in a commercial environment. This product generates, uses, and can radiate radio frequency energy, and if not installed and used in accordance with the instruction manual, might cause harmful interference to radio communications. Operation of this product in a residential area is likely to cause harmful interference, in which case you will be required to correct the interference at your own expense.



| |
|----------------|
| WARNING |
|----------------|

Changes or modifications not expressly approved by the party responsible for compliance could void the user's authority to operate the product.

FCC Supplier's Declaration of Conformity

Hereby declares that the product,

Product name: BTX III X-ray Diffraction Analyzer

Model: BTX III

Conforms to the following specifications:

FCC Part 15, Subpart B, Section 15.107 and Section 15.109.

Supplementary information:

This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions:

- (1) This device may not cause harmful interference.
- (2) This device must accept any interference received, including interference that may cause undesired operation.

Responsible party name:

Olympus Scientific Solutions Americas Corp.

Address:

48 Woerd Avenue, Waltham, MA 02453, USA

Phone number:

+1 781-419-3900

ICES-001 (Canada) Compliance

This Class A digital apparatus complies with Canadian ICES-001.

Cet appareil numérique de la classe A est conforme à la norme NMB-001 du Canada.

Code de la santé publique (France)

Conformément aux articles L. 1333-4 et R. 1333-17 du Code de la santé publique, l'utilisation ou la détention de ces analyseurs sont des activités soumises à autorisation de l'Autorité de sûreté nucléaire.

Packing and Return Shipping

If the BTX III is not returned in its transport case, it could be damaged during shipping. Olympus reserves the right to void the warranty on instruments damaged while in transit if they are shipped without their transport case. Prior to returning any units, contact Customer Service to obtain the required RMA number(s) and any important shipping information.

Follow the steps below to return your BTX III:

1. Pack the BTX III back into the transport case that it came in using the original packing materials.
2. Include the RMA in the case, and reference the RMA number in your shipping documents.
3. Close the transport case and do at least one of the following:
 - Secure the case with plastic zip ties.
 - Pack the transport case within another box.

Open Source Software

This product may include (i) open source software; and (ii) other software whose source code is intentionally published (collectively, hereinafter referred to as “OSS”).

The OSS included in this product shall be licensed and distributed to you subject to the terms and conditions applied to the OSS. Please see such terms and conditions of the OSS at the following URL:

<https://www.olympus-ims.com/btx-terra-open-source-software/>

The copyright owners of the OSS are listed at the above URL.

THERE IS NO WARRANTY FOR THE OSS, TO THE EXTENT PERMITTED BY APPLICABLE LAW. THE OSS IS PROVIDED “AS IS” WITHOUT WARRANTY OF ANY KIND, EITHER EXPRESSED OR IMPLIED, INCLUDING, BUT NOT LIMITED TO, THE IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE. THE ENTIRE RISK AS TO THE QUALITY AND PERFORMANCE OF THE OSS IS WITH YOU. SHOULD THE OSS PROVE DEFECTIVE, YOU ASSUME THE COST OF ALL NECESSARY SERVICING, REPAIR, OR CORRECTION.

Some of the OSS licenses associated with this product may permit you to obtain source code for certain software which Olympus has an obligation to provide in accordance with the terms and conditions applied to the OSS. You may obtain a copy of this source code at the following URL. This offer is valid for a period of three (3) years from the date of original purchase. Olympus has no responsibility to provide any source code except the source code for certain software.

<https://www.olympus-ims.com/btx-terra-open-source-software/>

Olympus does not respond to any inquiries related to any of the source codes obtained at the above URL.

Warranty Information

Olympus guarantees your Olympus product to be free from defects in materials and workmanship for a specific period, and in accordance with conditions specified in the *Olympus Scientific Solutions Americas Inc. Terms and Conditions* available at <http://www.olympus-ims.com/en/terms/>.

The Olympus warranty only covers equipment that has been used in a proper manner, as described in this instruction manual, and that has not been subjected to excessive abuse, attempted unauthorized repair, or modification.

Inspect materials thoroughly on receipt for evidence of external or internal damage that might have occurred during shipment. Immediately notify the carrier making the delivery of any damage, because the carrier is normally liable for damage during shipment. Retain packing materials, waybills, and other shipping documentation needed in order to file a damage claim. After notifying the carrier, contact Olympus for assistance with the damage claim and equipment replacement, if necessary.

This instruction manual explains the proper operation of your Olympus product. The information contained herein is intended solely as a teaching aid, and shall not be used in any particular application without independent testing and/or verification by the operator or the supervisor. Such independent verification of procedures becomes increasingly important as the criticality of the application increases. For this reason, Olympus makes no warranty, expressed or implied, that the techniques, examples, or procedures described herein are consistent with industry standards, nor that they meet the requirements of any particular application.

Olympus reserves the right to modify any product without incurring the responsibility for modifying previously manufactured products.

Technical Support

Olympus is firmly committed to providing the highest level of customer service and product support. If you experience any difficulties when using our product, or if it fails to operate as described in the documentation, first consult the user's manual, and then, if you are still in need of assistance, contact our After-Sales Service. To locate the nearest service center, visit the Service Centers page at: <http://www.olympus-ims.com>.

Introduction

The Olympus BTX III is a benchtop X-ray diffraction (XRD) analyzer designed primarily for analyzing a variety of powder sample types. Phase identification is obtained by comparing the diffraction signature of a sample with a database of XRD mineral patterns.

The BTX III analyzer uses a low power X-ray source and 2-D charge-coupled device (CCD) detector to obtain XRD data.

The BTX III analyzer incorporates the following independent safety protection circuits:

- Power key switch — Key must be inserted and in the ON position to turn the system on.
- X-ray warning indicators — LEDs on the front panel illuminate when X-rays are being generated, and also during the power on sequence.
- Sample carrier safety interlock — High voltage from the power supply is interrupted and generation of X-rays ceases if the sample carrier is removed during system operation.

1. Analyzer Overview

This chapter provides an overview of the BTX III X-ray diffraction analyzer and its accessories.

1.1 Packing List

Table 2 on page 23 lists the BTX III components.

Table 2 BTX III components

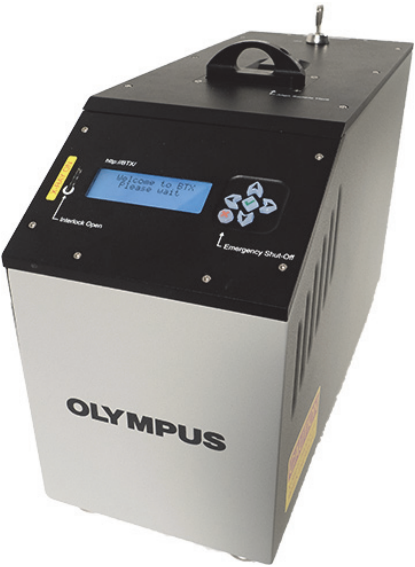
| | Component | BTX III – all models |
|---|------------------|--|
| 1 | BTX III analyzer |  |

Table 2 BTX III components (continued)



| Component | | BTX III – all models |
|-----------|--|--|
| 2 | 110 V/220 V AC-DC power supply #PWRS-10047/Q0201663 |  |
| 3 | External sample shaker assembly |  |

Table 2 BTX III components (continued)

| Component | | BTX III – all models |
|--|--|----------------------|
| Accessory kit (Plastic case Part #CASE-10033) | | |
| 1 | Micro spatula #TER11093 | |
| 2 | Ball driver #TOOL-10147 | |
| 3 | Power keys (2) | |
| 4 | USB flash drive (loaded with software, database, and documentation) #TER02200 | |
| 5 | Sample cells (1 Kapton and 1 Mylar) #TER11073/#TER11074 #TER11075 #TER11076/#WIND-10008 | |
| 6 | Sample cell screws (8) #TER11071 | |
| 7 | Sample crusher #LBSP-10008 | |
| 8 | Sample sieve #LBSP-10009 | |

1.2 Front/Top Panel

The front and top panels are where all BTX III controls, indicators, and the sample chamber are located (see on page 26 and Table 3 on page 27).



Figure 1-1 BTX III front panel

NOTE

Depending on your location, the device may have a secondary indicator light on the top of the unit.

Table 3 BTX III front/top panel items

| Item | Description | Item | Description |
|-------------|-------------------------------|-------------|--------------------|
| 1 | Power key switch | 4 | Sample chamber |
| 2 | Stop/Emergency Shutoff button | 5 | Keypad |
| 3 | LED indicators | 6 | Display |

1.2.1 Power Key Switch

Turn the power key clockwise to turn on the BTX III analyzer (see on page 28).

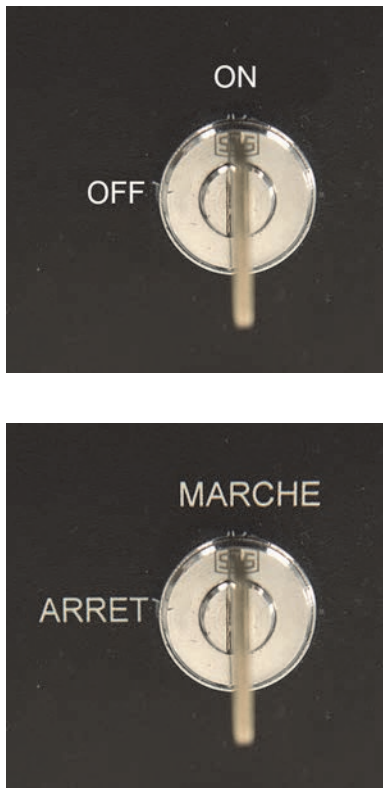


Figure 1-2 Power key switch (On)

NOTE

The power key text may display in English or French according to your location.

1.2.2 Keypad

The keypad enables you to make selections on the display to set up and run tests and save results.



Figure 1-3 Keypad

1.2.3 Stop/Emergency Shutoff Button

The Stop/Emergency Shutoff button enables you to stop an in-progress test, turn off system power in an emergency, or cancel a selection when using the keypad and main menu to set up tests (see Figure 1-4 on page 29).



Figure 1-4 Stop/Emergency Shutoff button

NOTE

Depending on your location, the button may not have accompanying text.

1.2.4 LED Indicators

The LED indicators show the status of the high-voltage power supply, X-ray tube, and safety interlocks (see on page 30).

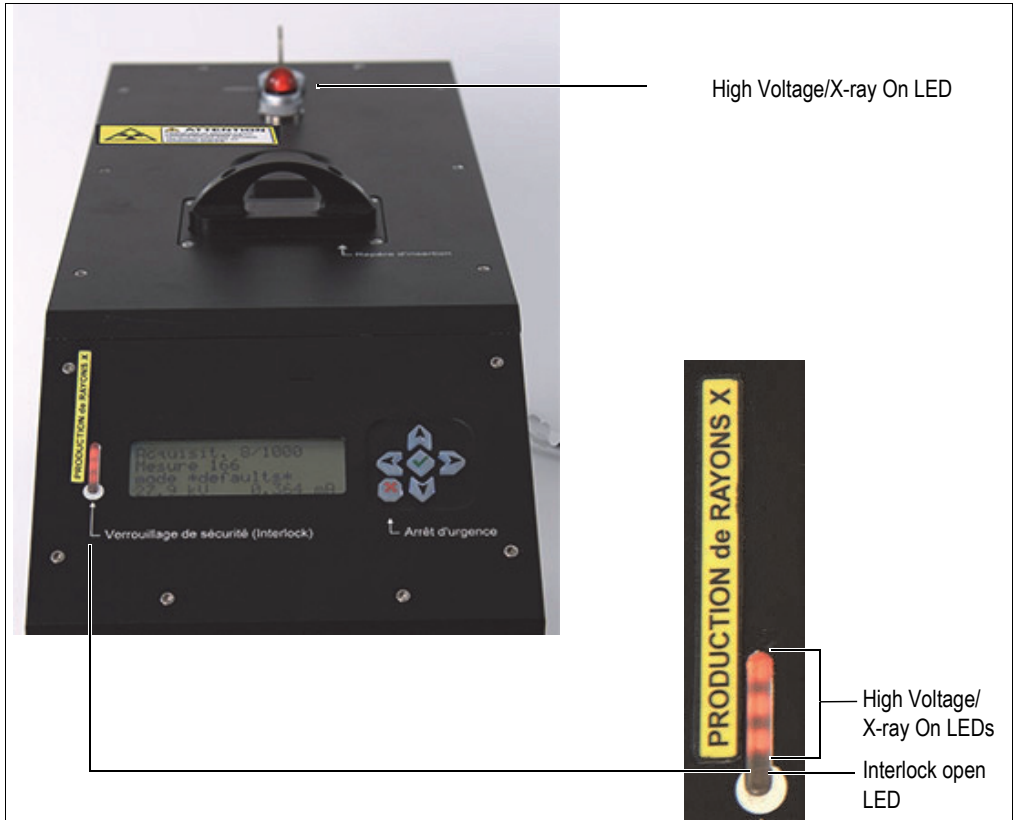


Figure 1-5 LED indicators

NOTE

Depending on your location, the device may have a secondary indicator light on the top of the unit.

1.2.5 Sample Chamber

The sample chamber is located in the center of the top panel. The clamp lever locks and unlocks the sample carrier (see Figure 1-6 on page 31).

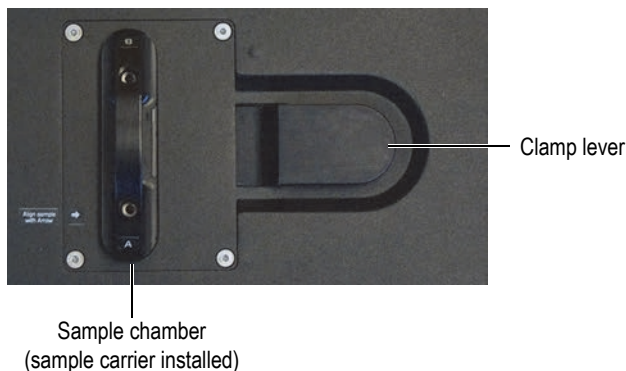


Figure 1-6 Sample chamber assembly

NOTE

The Sample Chamber text may display in English or French according to your location.

1.2.6 Display

The display shows selections for setting up and monitoring tests (see Figure 1-7 on page 31).



Figure 1-7 Display

NOTE

The menu text may display in English or French according to your location.

1.3 Rear Panel

The rear panel is where all BTX III connectors are located (see Figure 1-8 on page 32 and Table 4 on page 33).

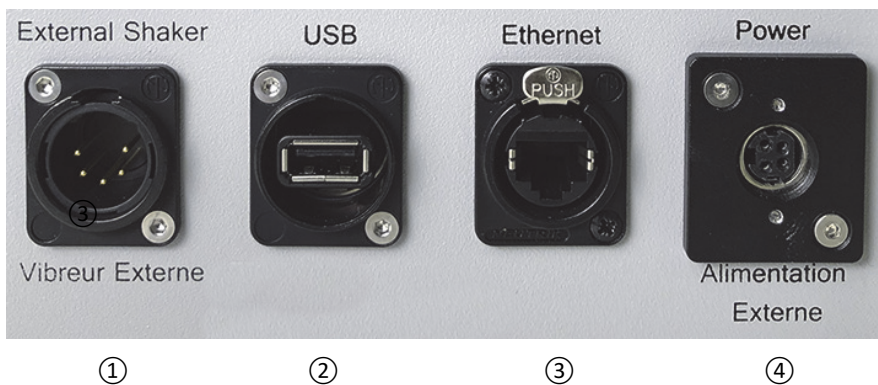


Figure 1-8 BTX III rear panel

Table 4 BTX III rear panel connectors

| Item | Description | Item | Description |
|-------------|--|-------------|--|
| 1 | External Shaker — Port for connecting the external sample shaker. The shaker is used to load and unload samples from the BTX III sample carrier. | 3 | Ethernet — Port for connecting the BTX III to an Ethernet network. |
| 2 | USB — Port for connecting a USB flash drive to transfer files to or from the BTX III analyzer. | 4 | Power — Port for connecting the BTX III analyzer to external AC power. |

2. Safety Information

This chapter contains important safety information for using the BTX III analyzer.

2.1 Radiation Safety Information

The fundamental principle in radiation protection is that all radiation exposures should be maintained as low as reasonably achievable (ALARA). This is referred to as the ALARA principle. The three key factors which influence an individual's radiation dose from a given source are time, distance, and shielding. Controlling these factors is the key to keeping the radiation dose ALARA.

- Time

The most direct way to reduce radiation dose is to reduce the time spent working with or in the vicinity of radiation sources. If the exposure time is cut in half, the dose will be reduced by the same fraction.

- Distance

Distance can effectively reduce a radiation dose. When the working distance from the radiation source is increased by a factor of two, the dose received from that source is reduced by a factor of four. This is referred to as the inverse square law, which states that the radiation intensity from a point source decreases by the square of the distance from the source.

- Shielding

Shielding is any material used to reduce the intensity of radiation by absorbing or attenuating the radiation coming from the source.



WARNING



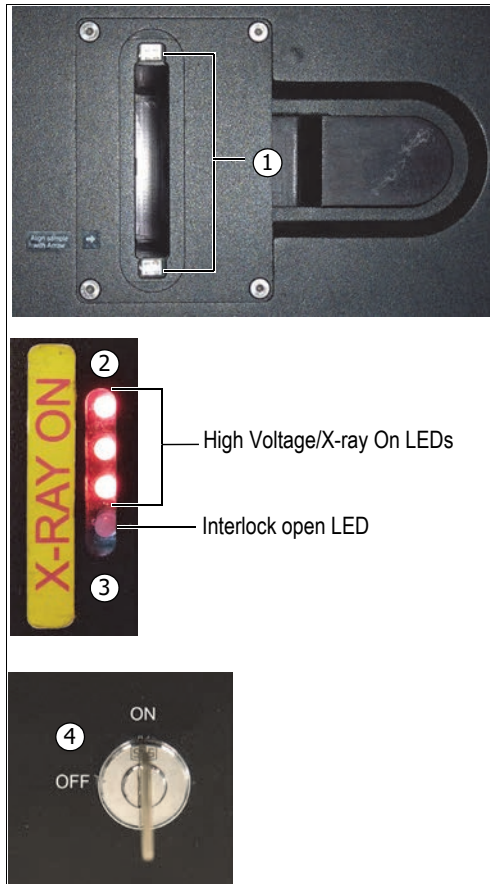
Do not open the system, disassemble, or modify any internal components. These actions could result in serious damage to the system and a health hazard to the operator.

2.2 Safety Interlocks

The BTX III analyzer uses an X-ray tube that produces ionizing radiation at up to 30 keV at very low power (10 W) as compared to laboratory XRD systems (typically greater than 1 kW). The BTX III system is designed with internal X-ray shielding to fully protect operators and internal components.

The radiation producing components are completely contained within the system housing and constructed in such a way that no measurable radiation is detected during operation. No beam alignment nor X-ray beam calibration by the operator is required. There is no reason for any BTX III operator to bypass any radiation safety switches. The BTX III analyzer fully complies with the FDA CFR, section 1020.40 including safety interlocks and radiation measurements. The BTX III analyzer has no measurable radiation leakage from any surface during operation. This is because of the shielding of the X-ray generation and detection components, and the low operating potential and power of the X-ray generation source.

The BTX III analyzer incorporates several independent safety interlock circuits to protect the operator. See Figure 2-1 on page 37 and Table 5 on page 38.

**NOTE**

Depending on your location, the device may have a secondary indicator light on the top of the unit, and labels may be in French or English.

Table 5 BTX III radiation safety features

| Label | Safety feature |
|-------|---|
| 1 | <p>Sample chamber interlock</p> <p>The X-ray beam only operates when the sample carrier is properly inserted into the system. If the carrier is removed while the system is in operation, the high-voltage power supply interrupts and X-ray generation ceases.</p> |
| 2 | <p>High voltage/X-ray On LEDs</p> <p>These LEDs glow red whenever high voltage is enabled and X-ray generation is possible.</p> |
| 3 | <p>Interlock open LED</p> <p>This LED is normally off. It glows amber when a safety interlock switch has been opened. A safety interlock switch could be triggered by removing the sample carrier during a sample analysis.</p> |
| 4 | <p>Power key switch</p> <p>The power key switch has ON and OFF positions. The key must be inserted and in the ON position for the system to operate, including operation of the X-ray beam.</p> |
| 5 | <p>Control panel interlock (not shown)</p> <p>This is a magnetic switch that prevents the operation of high voltage when the control panel (top panel) is improperly seated or removed from the outer case. Under these conditions, the X-ray beam will not operate.</p> |

Figure 2-2 on page 39 shows the LEDs described in Table 5 on page 38. Table 6 on page 40 describes the behavior of the LEDs in relation to X-ray emissions.

| |
|-------------|
| NOTE |
|-------------|

Depending on your location, the device may have a secondary indicator light (LED 3 in Table 6 on page 40) on the top of the unit, and labels may be in French or English.

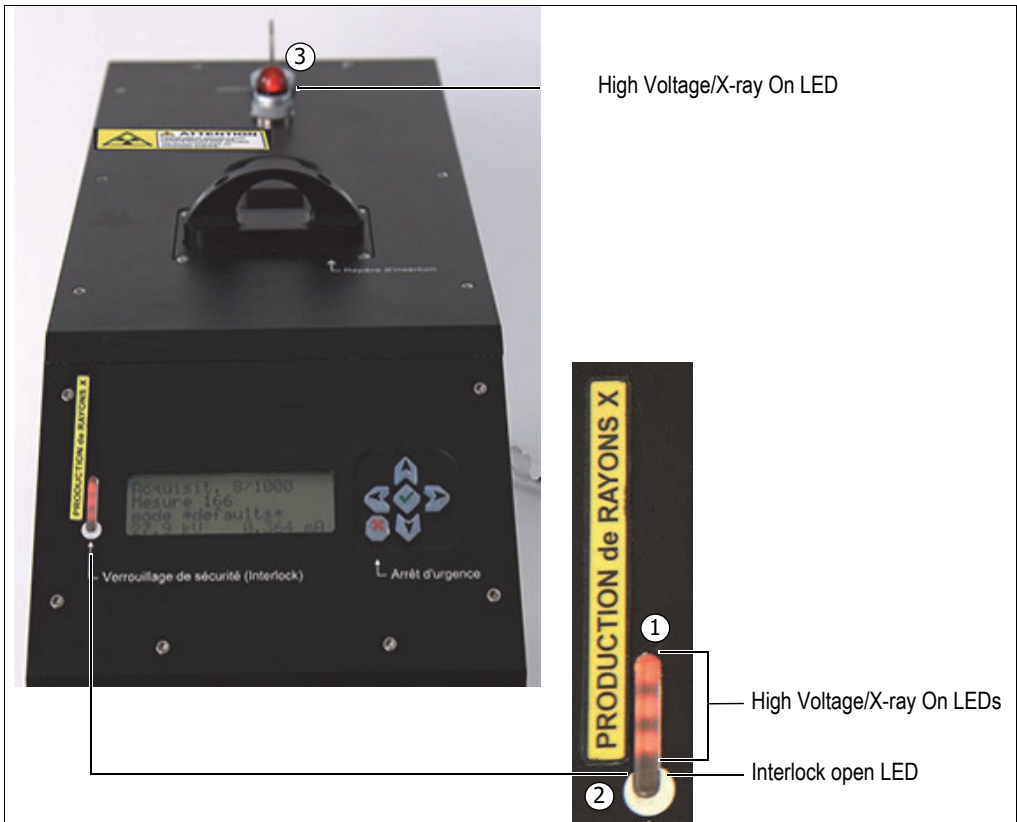


Figure 2-2 BTX III LED indicators

2.3 Radiation Dose Measurements

Table 6 LED behavior

| Case | High voltage | X-ray | LED 1 (red) | LED2 (amber) | LED 3 (red) |
|----------------|--------------|------------------------|----------------|--------------|----------------|
| 1 | On | On | On | Off | On |
| 2 | Off | Off | LED FAIL (Off) | Off | Off |
| 3 | Off | Off | Off | Off | LED FAIL (Off) |
| 4 ^a | On | FILAMENT FAILURE (Off) | On | Off | On |
| 5 | Off | INTERLOCKS OPEN (Off) | Off | On | Off |

- a. A current drop during a measurement can be related to a filament failure. In such a case, please contact the Olympus After-Sales Service. If the filament fails, the X-ray automatically turns off while the high voltage remains active. The X-ray tube is specifically designed to withstand such a breakdown and the electrical safety is maintained.

Radiation dose measurements were made to document any possible ionizing radiation dose to a typical operator of the BTX III XRD analyzer. The measurements were made using a calibrated Ludlum Model 9-3 Radiation Ion Chamber. This chamber is capable of measuring low energy X-ray fields to within plus or minus 20 % of true value above 10 keV, with a typical counting range of 0 $\mu\text{C}/\text{kg}$ to 51.6 $\mu\text{C}/\text{kg}$ (0 mR/hr to 200 mR/hr).

During testing, the BTX III analyzer was operated at X-ray tube conditions that are standard for all testing materials (30 kV, 360 μA in France, 330 μA in the rest of the world). Radiation dose measurements were made at specific locations at the base, back, front, and sides of the system.

Radiation dose measurements around the perimeter of the system, at a distance of less than 2 cm, yielded no measurable radiation levels (less than 0.2 mR/h). This level is well within acceptable levels of exposure to the general public. When properly set up and operated, there is no exposure in excess of the typical dosage to the general public of naturally occurring sources of ionizing radiation.

Figure 2-3 on page 41 shows the radiation dose measurement points. The radiation levels measured at these points are reported in Table 7 on page 42. All measurements were made at a maximum power setting of 30 kV and 360 μA in France (330 μA in the rest of the world).



Figure 2-3 Radiation measurement points

| |
|-------------|
| NOTE |
|-------------|

Depending on your location, the device may have a secondary indicator light on the top of the unit.

Table 7 Radiation level measurements

| Measurement point | Measured radiation level in $\mu\text{C}/\text{kg}$ (R/hr) | Distance from surface (cm) | Comments |
|--------------------------|--|-----------------------------------|--------------------|
| A | < 0.0129 (0.00005) | 2 | Front top panel |
| B | < 0.0129 (0.00005) | 2 | Left side of case |
| C | < 0.0129 (0.00005) | 2 | Front side of case |
| D | < 0.0129 (0.00005) | 2 | Right side of case |
| E | < 0.0129 (0.00005) | 2 | Back side of case |
| F | < 0.0129 (0.00005) | 2 | Bottom of case |

3. Set Up and Operation

This chapter provides information for turning on and off the BTX III XRD analyzer and preparing and testing samples.

3.1 Connecting AC Power to the BTX III

The BTX III analyzer receives AC power through the External Power connector.

To connect AC power



CAUTION

If an unauthorized power supply cord is used to power the instrument, Olympus cannot guarantee the electrical safety of the equipment.

1. Hold the power supply output plug in the orientation shown, and then insert it into the rear panel Power connector (see Figure 3-1 on page 44).



Figure 3-1 Power plug and connector

2. Plug one end of the AC power cord firmly into the power supply (see Figure 3-2 on page 44).



Figure 3-2 Power supply

3. Plug the other end of the AC power cord into a live AC (mains) power outlet.

3.2 Turning On or Off the BTX III

To turn on the BTX III

- ◆ Turn the power key switch clockwise to the ON position (see on page 28).

The system briefly displays the message:

Welcome to BTX---Booting


Please wait

After about a minute, the display reads:

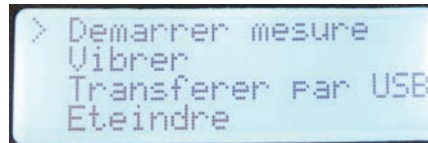
Cooling to: -45

Current temp: (temperature)

After the detector cools to -45°C , the menu appears (see Figure 3-3 on page 45).



```
> Start acquisition
Shake
Do USB transfer
Shut down
```



```
> Demarrer mesure
Vibrer
Transferer par USB
Eteindre
```


Figure 3-3 Menu

| |
|-------------|
| NOTE |
|-------------|

Depending on your location, the menu may be in French or English.

To turn off the BTX III in an emergency situation

- ◆ Turn the power key switch to OFF.
OR

Press the **Stop/Emergency Shutoff** button () twice (quickly).

To turn off the BTX III under normal conditions

1. On the menu, select **Shut Down** to power the unit off.
The display reads:
Shutting down...
Please wait...
2. After the unit is off, turn the power key switch to OFF.

3.3 Preparing a Sample for Analysis

BTX III analyzer is designed to operate with coarsely ground samples. The samples must be dry and be able to pass through a 150 μm sieve. Also the sample size should be large enough to convect within the sample cell. Very small particles generally do not perform well within a standard BTX III sample cell. The particles tend to stick to one another and not convect as they should. If you encounter this condition, contact Olympus for details on either increasing the sample cell volume, or to purchase an alternate sample cell.

To prepare a sample for analysis

1. Crush the sample in a crusher (see Figure 3-4 on page 47).
The result is a sample with a variety of particle sizes.



Figure 3-4 Crushing a sample

2. Refine the sample further using a sample sieve (see Figure 3-5 on page 47).



Figure 3-5 Sample sieves

3.4 Loading a Sample

Before you can load a crushed sample into the BTX III analyzer, you must remove the sample carrier from the instrument, then load the sample into the sample cell.

There are four components of a sample cell (see Figure 3-6 on page 48):

- An inner cell window comprised of a polymer window on a metal frame with a notch at the top
- A spacer, which is assembled between the inner and outer cell
- An outer cell window that also has a polymer window on a metal frame, but without a notch at the top
- Screws to secure the cell assembly to a sample cell carrier

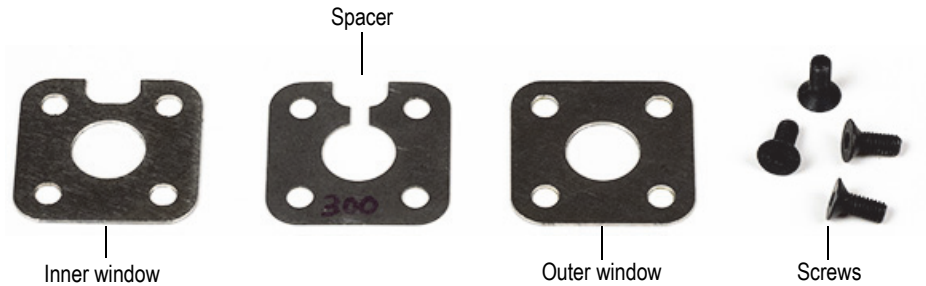


Figure 3-6 Sample cell window components

The BTX III analyzer has a sample carrier that can accommodate two cell assemblies. Only side A should be used when loading a cell. Side B is provided as a counter mass for vibration and as a spare cell (see Figure 3-7 on page 48).

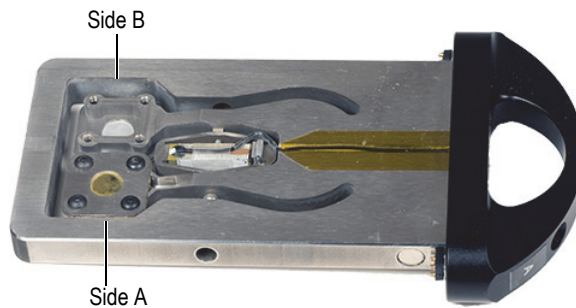


Figure 3-7 Sample carrier

The BTX III analyzer is supplied with an external shaker assembly to ease sample loading and unloading (see Figure 3-8 on page 49). The external shaker assembly is used in conjunction with the sample cell carrier to “shake” or vibrate the sample into the cell assembly.



Figure 3-8 External shaker assembly

To remove the sample carrier

1. Lift the lever to unlock the sample carrier (see Figure 3-9 on page 49).

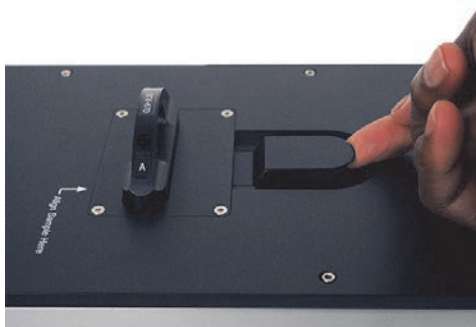


Figure 3-9 Unlocking the sample carrier

2. Pull up the carrier handle to remove the sample carrier (see Figure 3-10 on page 50).



Figure 3-10 Removing the sample carrier

To assemble a cell

1. Lay the inner window down first (see Figure 3-11 on page 50).
Make sure that the polymer film faces up.
2. Place the spacer on top of the inner window.
3. Place the outer window on top of the spacer.
Make sure that the polymer film faces the spacer.

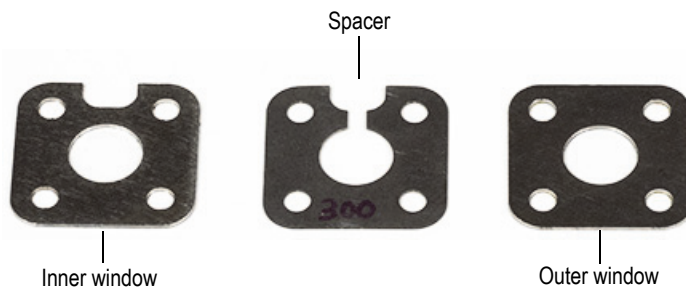
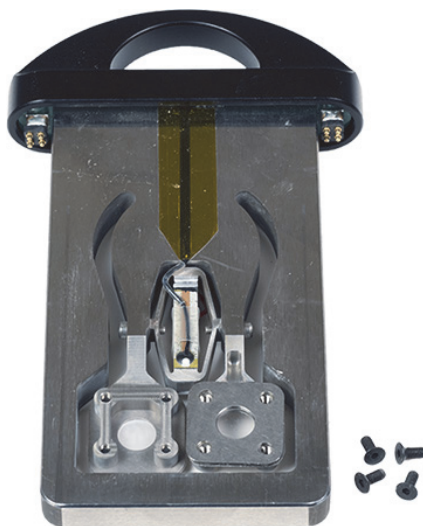


Figure 3-11 Sample cell window components

4. Align the windows and spacer so that the holes in the cell assembly are aligned with the holes in the sample carrier (see Figure 3-12 on page 51).
5. Replace the holding screws, being very careful not to puncture the polymer window material.
6. Tighten the holding screws.



Cell windows aligned and stacked in proper sequence

Figure 3-12 Sample cell ready for screws

To load a sample into a sample cell

1. Connect the external shaker plug to the External Shaker connector on the rear panel (see Figure 3-13 on page 51).

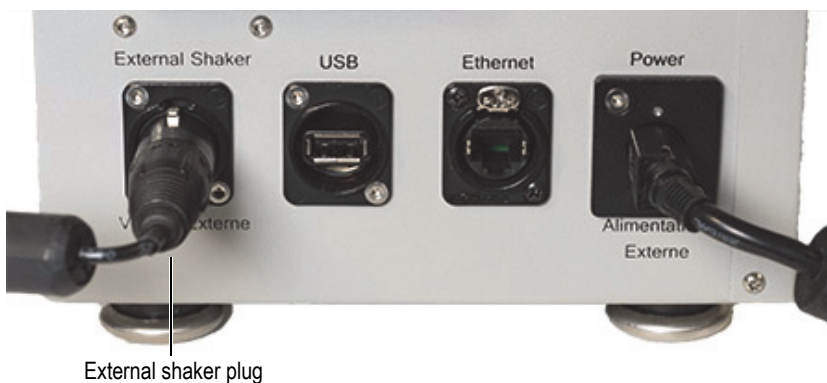


Figure 3-13 External shaker plug connected

2. Insert the sample carrier into the external shaker and gradually load a small amount of material (enough to fill the gap created by the spacer) into the side A cell assembly (see Figure 3-14 on page 52).
The material should be approximately 50 mg of coarsely ground (100–150 μm) powder.

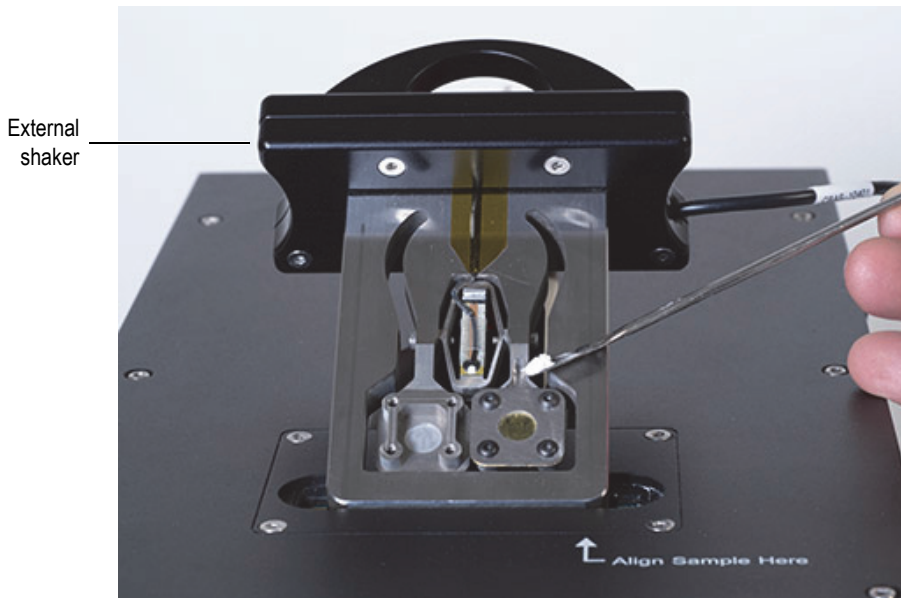







Figure 3-14 Loading the sample

3. On the main menu, select **Shake**, then press the Check button ().
The external shaker vibrates to aid in loading the sample.
4. Adjust the shaking amplitude using the Left arrow () and Right arrow () buttons.
5. To save the selected shaking amplitude value, press the Check button ().
6. When the cell appears to be full, press the Stop button (), and then remove the sample carrier from the external shaker.

3.5 Testing a Sample

To test a sample

1. Insert the sample carrier into the sample chamber, making sure that side A is oriented toward the right side of the system (see Figure 3-15 on page 53).



Figure 3-15 Inserting the sample carrier

2. Lock the sample carrier in place by pushing the lever down (see Figure 3-16 on page 54).

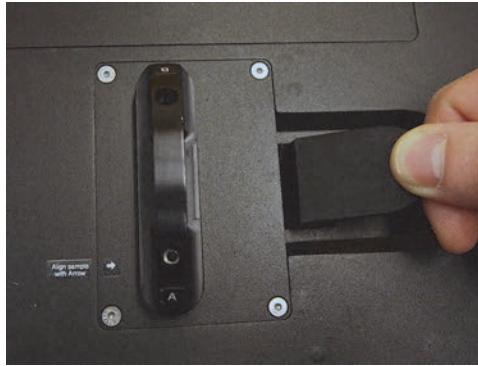


Figure 3-16 Locking the sample carrier down


3. Select **Start acquisition** in the display, then select a running mode (see Table 8 on page 54).

Table 8 Running modes

| Menu level one | Menu level two | Level two choice | Comments |
|--------------------------|--------------------|---------------------|---|
| Start acquisition | | | |
| | Choose Mode | | |
| | | *Default* | Uses the default test mode that has been set up by the administrator in the SwiftMin® software (see “Mode Setup Tab” on page 76). |
| | | Custom modes | Uses a test mode that has been set up by the administrator in the SwiftMin® software (see “Mode Setup Tab” on page 76). |
| Shake | | | |

Table 8 Running modes (continued)


| Menu level one | Menu level two | Level two choice | Comments |
|------------------------|-----------------------------|-------------------|--|
| | Shaking X to quit | | Initiates vibration of the external shaker assembly, assuming it is connected. Press the Stop button on the keypad to terminate external shaking. Adjust the shaking amplitude with the left and right arrows. Save this value with the Check key. |
| Do USB Transfer | | | |
| | Choose Dataset | (Data set) | Selects the data set that is transferred to the USB flash drive. |
| Shut Down | | | Turns off the BTX III analyzer. The system takes a few minutes to slowly warm up the detector, preventing condensation from forming and avoiding damage. |

4. Press the Check button () to start the test.

When the test begins, the BTX III analyzer gives the data set a name based on a sequential number. The red LEDs glow, indicating the application of high voltage to the X-ray tube. The display shows the voltage and intensity readings, and the sample carrier emits a high-pitched vibration noise.

NOTE In some locations you can only start tests using the Check button on the front panel keypad. Tests cannot be started through the BTX III SwiftMin® software user interface.

To stop testing before all the exposures are taken

- ◆ Press the Stop button () once.

3.6 Unloading a Sample

To remove the sample carrier from the sample chamber

1. Lift the lever to unlock the sample carrier.
2. Lift the sample carrier out of the sample chamber.

To unload a sample from the sample cell

- ◆ Remove the majority of the material with the aid of the external shaker assembly.

OR

1. Using a 1.5 mm hex driver, carefully remove the holding screws, and then disassemble the cell windows and spacer (see Figure 3-17 on page 56).
2. Clean or replace the cell parts as necessary.



Figure 3-17 Cell disassembly

4. Local Area Network Communication

You must establish a local area network (LAN) connection between your PC, tablet, or other device and the BTX III analyzer before you can use a web browser to run the SwiftMin® software (see “SwiftMin® Software User Interface” on page 65).

You can set up the BTX III to communicate with a LAN using one of the two following methods:

- Connect an Ethernet cable to the Ethernet connector to establish a wired LAN.
- Connect a wireless LAN dongle to the USB connector to establish a wireless LAN. Your PC, tablet, or other device must be wireless LAN (WLAN) enabled with a compliant 802.11b/g/n connection.

The device you use to communicate with the BTX III must be capable of supporting the full functionality of the SwiftMin® software.

| |
|-------------|
| NOTE |
|-------------|

Your LAN must be capable of connecting using Dynamic Host Configuration Protocol (DHCP). If your network does not recognize DHCP, contact your IT department for the proper addresses.

4.1 Checking the Network Configuration

NOTE

The BTX III can connect to either a wired LAN or broadcast a wireless LAN, but not both at the same time. The wired LAN connection is the default if an Ethernet cable and a wireless LAN dongle are both connected to the BTX III at the same time.

To check the network configuration

NOTE

The default network setting of the BTX III is **WIRED ON** (Ethernet).

1. Make sure the BTX III is on and the Main menu is displayed.
2. Briefly press both the Left arrow (◀) and Right arrow (▶) buttons at the same time to display the Advanced menu (see Figure 4-1 on page 58).
3. Press the Down arrow button (▼) to select **Configure Network**.



Figure 4-1 Advanced menu

4. Press the Check button (◊) to confirm the selection and display the DHCP (Dynamic Host Configuration Protocol) menu (see Figure 4-2 on page 59).
 - If the LAN is already set to **WIRED ON**, the BTX III is configured to connect to your LAN through the Ethernet connector.

See “Broadcasting a WLAN through a Wireless LAN Dongle” on page 60 for instructions on setting **WIRED** to **OFF**.

- If the LAN is set to **WIRED OFF**, the BTX III is configured to broadcast a WLAN through a wireless LAN dongle.

See “Connecting to your LAN through the Ethernet Connector” on page 59 for instructions on setting **WIRED** to **ON**.

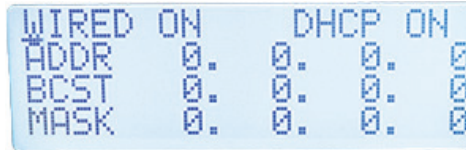





Figure 4-2 DHCP menu

4.2 Connecting to your LAN through the Ethernet Connector

When the LAN is set to **WIRED ON**, the BTX III is configured to connect to your LAN through the Ethernet connector.

To connect to your LAN through the Ethernet connector

1. Connect an Ethernet cable to the Ethernet connector on the rear of the BTX III.
2. Use the arrow buttons to set **WIRED** to **ON**:
 - a) Use the right arrow button () to move from the **WIRED** field to the **OFF** field.
 - b) Use the up or down arrow button ( ) to change the value to **ON**.
3. To quickly set up the protocol, use the arrow buttons to set **DHCP** to **ON** (see Figure 4-3 on page 60). Otherwise, change the DHCP parameters using the arrow buttons.

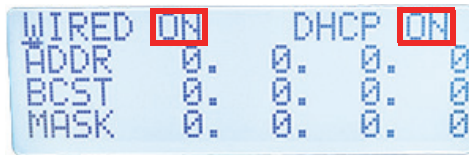



Figure 4-3 DHCP menu

4. Press the **Check** button () to accept the changes and restart the BTX III. The BTX III restarts with the new DHCP configuration.
5. After the BTX III has restarted, navigate back through **Main Menu > Advanced Menu > Configure Network > DHCP Menu** to view the network-assigned IP address (see Figure 4-4 on page 60).

| |
|-------------|
| NOTE |
|-------------|

The network-assigned IP address must be used in your Web browser to access the BTX III software.

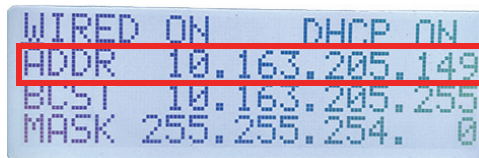


Figure 4-4 Network-assigned IP address

4.3 Broadcasting a WLAN through a Wireless LAN Dongle

When the LAN is set to **WIRED OFF**, the BTX III is configured to broadcast a WLAN through a wireless LAN dongle.

To broadcast a WLAN through the wireless LAN dongle

1. Connect a wireless LAN dongle to the rear panel USB port.

2. Use the arrow buttons to set **WIRED** to **OFF**:





- Use the right arrow button () to move from the **WIRED** field to the **ON** field.
- Use the up or down arrow button ( ) to change the value to **OFF** (see Figure 4-5 on page 61).



Figure 4-5 DHCP menu

3. Press the **Check** button () to accept the changes and restart the BTX III. The BTX III restarts with the new DHCP configuration and begins broadcasting a WLAN.

4.4 Connecting your PC to the WLAN

Through the wireless LAN dongle, the BTX III broadcasts an unsecured WLAN that is identified by the serial number of the analyzer. For example, if the serial number of your BTX III is “670,” then it broadcasts on a network identified as “BTX-670.”

To connect your PC to the WLAN

1. Turn on your WLAN-configured BTX III and wait until the menu is displayed (see Figure 4-6 on page 62).



Figure 4-6 Menu

NOTE

Depending on your location, the menu may be in French or English.

In your PC available networks menu, find the serial number associated with your BTX III (see Figure 4-7 on page 62).

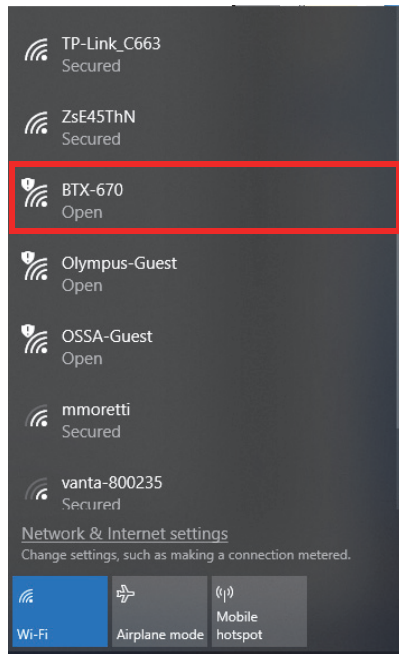


Figure 4-7 BTX III network broadcasting

2. Connect your PC to the BTX III (see Figure 4-8 on page 63).

You are now ready to set up and run tests using the SwiftMin® software (see “SwiftMin® Software User Interface” on page 65).

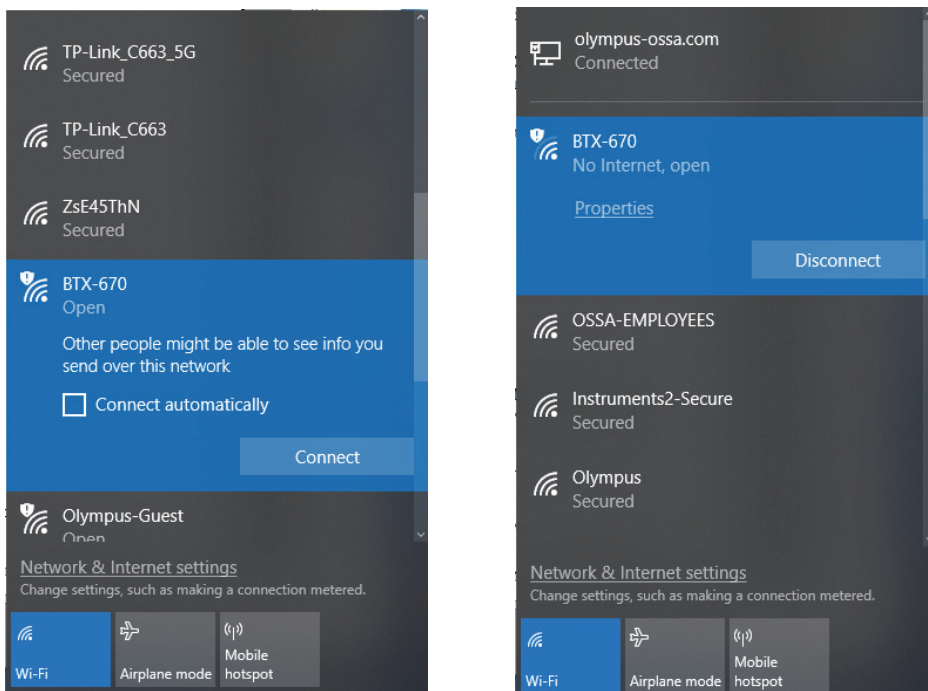


Figure 4-8 BTX III network selected (left) and connected (right)

5. SwiftMin® Software User Interface

The SwiftMin® software user interface (UI) enables you to set up and run tests on the BTX III X-ray diffraction analyzer.

| |
|-------------|
| NOTE |
|-------------|

The web browser that you use to run the SwiftMin® software must be capable of supporting the full SwiftMin® functionality.

5.1 Opening and Closing the SwiftMin® Software User Interface

The SwiftMin® software UI runs in a web browser of your choice.

To open the SwiftMin® software user interface

1. Start a web browser on your device.
2. Type the network-assigned IP address displayed on the DHCP menu into the browser address bar if using Ethernet, or type **http://192.168.0.222** into the browser address bar if using the WLAN.

This initiates a connection with the SwiftMin® software and displays the UI (see Figure 5-1 on page 66).

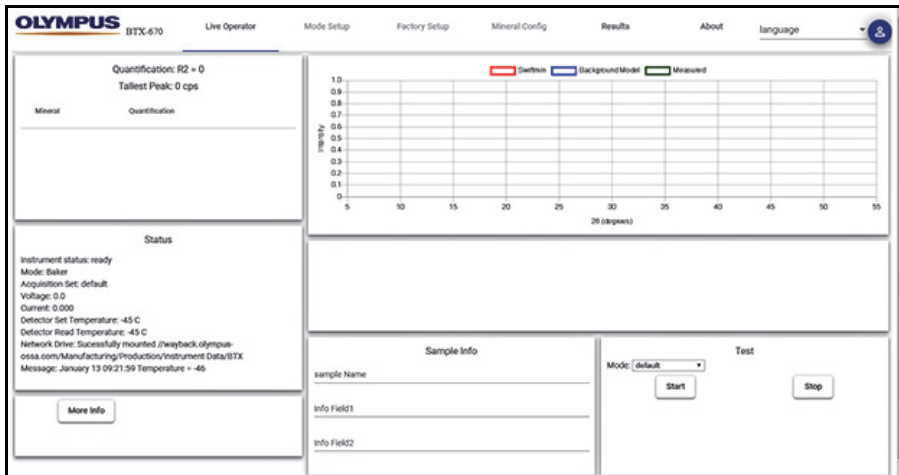


Figure 5-1 SwiftMin® software user interface

To close the SwiftMin® software user interface

- ◆ In your web browser, close the **XRDA** tab.

5.2 SwiftMin® User Access Levels

The SwiftMin® software provides two user access levels:

- Default
- Manager

The Default access level is used for real-time operation of the BTX III analyzer. The Manager access level also provides real-time operational capability, and can also be used to configure the mineral databases and test parameters of the instrument.

NOTE

To acquire the password for the Manager login, contact your Olympus sales representative.

5.2.1 Default Access

Default access is available when the software starts up. No password is required. Three screens are available:

- Live Operator
- Results
- About

| |
|-------------|
| NOTE |
|-------------|

Screen names that you cannot access are grayed out.

The **Live Operator** screen is displayed when the software starts up. The **Results** and **About** screens are selectable.

5.2.2 Manager Access

Manager access is password protected. Five screens are available:

- Live Operator
- Mode Setup
- Mineral Config
- Results
- About

| |
|-------------|
| NOTE |
|-------------|

The Factory Settings screen is grayed out.

To log in with Manager access

1. Click the icon in the upper-right corner of the screen (see Figure 5-2 on page 68).

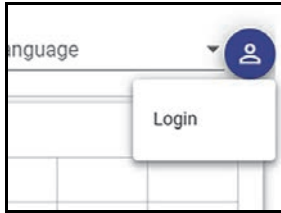


Figure 5-2 Manager access icon

2. In the dialog box, click **Login**.
3. Enter the password in the **Enter Password** dialog box, and click **Ok** (see Figure 5-3 on page 68).

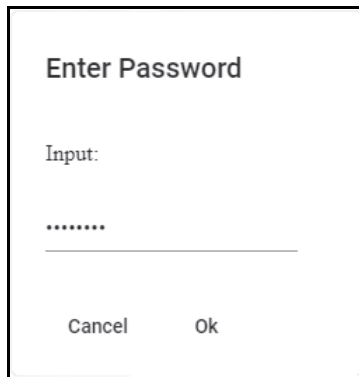


Figure 5-3 Enter Password dialog box

To log out of Manager access

1. Click the icon in the upper-right corner of the screen.
2. In the dialog box, click **Logout**.

5.3 Using the SwiftMin® Software

The software workflow depends on your access level.

To change the current screen

- ◆ In the menu bar, click the tab of the screen you want to display (see Figure 5-4 on page 69).



Figure 5-4 Menu bar

5.3.1 Screen Tabs

The screen tabs enable you to access the workflows.

- **Live Operator**
Used to start or stop a test and to check the status of the instrument.
- **Mode Setup** (not available with default access)
Used to add, edit, and delete modes. A mode includes test parameters such as the SwiftMin® and SwiftMin® RIR databases, number of exposures, piezo volume, and network parameters.
- **Mineral Config** (not available with default access)
Used to quickly edit SwiftMin® RIR databases, and upload or download SwiftMin® RIR databases as .csv files for more extensive editing.
- **Results**
Used to view, review, or download current or past results and data. Can also set the calibration.
- **About**
Displays version and licensing information about the SwiftMin® software.

5.3.2 Default Access Workflow

This workflow enables you to set up and run a test, then download selected results. You can view and download the current result or previous results.

To run a test

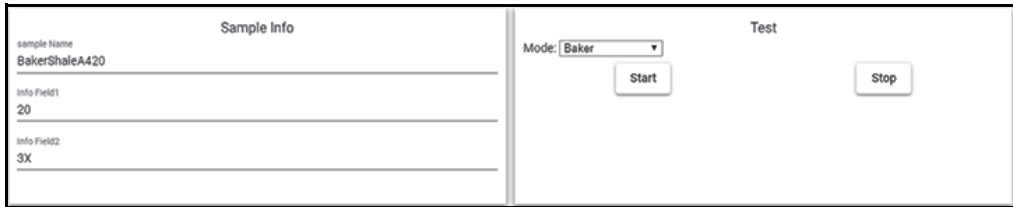
1. In **Test** dialog box of the **Live Operator** screen, select a test mode (see Figure 5-5 on page 70).
2. In the **Sample Info** dialog box, enter a **Sample Name** (30 alphanumeric characters maximum).

Optionally enter information into **Info Field1** (10 characters maximum) and/or **Info Field2** (4 characters maximum).

The information that you enter in the **Sample Info** dialog box can be used to uniquely identify the test in the Results screen.

For example, if you enter “BakerShaleA420” in **Sample Name**, “20” in **Info Field1**, and “3X” in **Info Field2**, the test name will appear in the results data as “BakerShaleA420_20_3X”

3. In **Test** area, click **Start**.



The screenshot shows two side-by-side panels. The left panel, titled "Sample Info", contains three input fields: "sample Name" with the text "BakerShaleA420", "Info Field1" with the text "20", and "Info Field2" with the text "3X". The right panel, titled "Test", features a dropdown menu labeled "Mode:" with "Baker" selected, a "Start" button, and a "Stop" button.

Figure 5-5 Test and Sample Info areas

NOTE

In some locations you can only start tests using the Check button on the front panel keypad. Tests cannot be started through the BTX III SwiftMin® software user interface.

The test will run until complete. Results are displayed as the test progresses.

To stop a test

- ◆ Click **Stop** (see Figure 5-5 on page 70).

To search for a result

1. Click the **Results** tab.
2. In the **Date/Mode** dialog box (see Figure 5-6 on page 71), use the calendar to find results by date (optional).
Use the **Start date** and/or **End date** choices to select a date range.

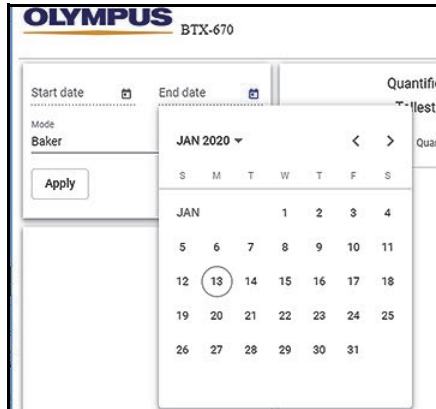


Figure 5-6 Date/Mode dialog box

3. Select a mode.
If you do not select a mode, the last selected mode is used.
4. Click **Apply**.
5. In the list that appears, choose the date and result that you want to display (see Figure 5-7 on page 72).

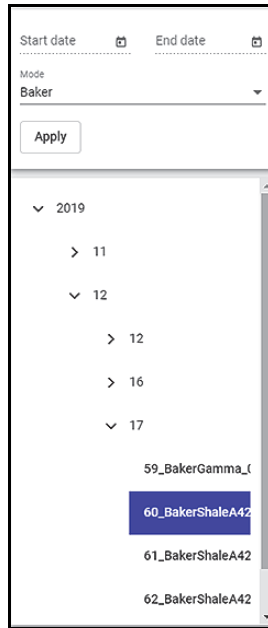


Figure 5-7 Result selected in list

To download the result data

- ◆ Click **Download Data** to download the results data to your PC.

To download the results

- ◆ Click **Download Results** to save the results only for the highlighted sample to the BTX III analyzer.

5.3.3 Manager Access Workflow

Manager access includes all of the Default access level functionality, and you can also configure mineral databases and test parameters of the instrument.

The BTX III manager can select a SwiftMin® reference intensity ratio (RIR) mineral database to edit within the SwiftMin® UI, and then save that database for later selection. The BTX III manager can also upload and download the SwiftMin®

American Mineralogist Crystal Structure Database (AMCSD) (.txt) and SwiftMin® RIR mineral databases (csv). This feature is useful to make extensive edits to existing RIR databases.

The BTX III manager can also add, edit, or delete test modes.

5.3.3.1 Mineral Config Tab

This tab enables you to configure mineral databases.

To work with the .csv mineral database

1. Click the **Mineral Config** tab.
2. Click the down arrow in the **SwiftMin DB** selection box, and choose a database (see Figure 5-8 on page 74).
After you select a database, the mineral list for that database is displayed.
3. In the mineral list, click the check box of the mineral that you want to enable or disable in the database (see Figure 5-8 on page 74).
4. Click **SaveAs** to save the database with a new or edited name.
The database is saved to the hard drive on the BTX III analyzer.

| |
|-------------|
| NOTE |
|-------------|

To avoid confusion, it is good practice to save the database with a new or edited name (by clicking **SaveAs**), indicating that a change was made.

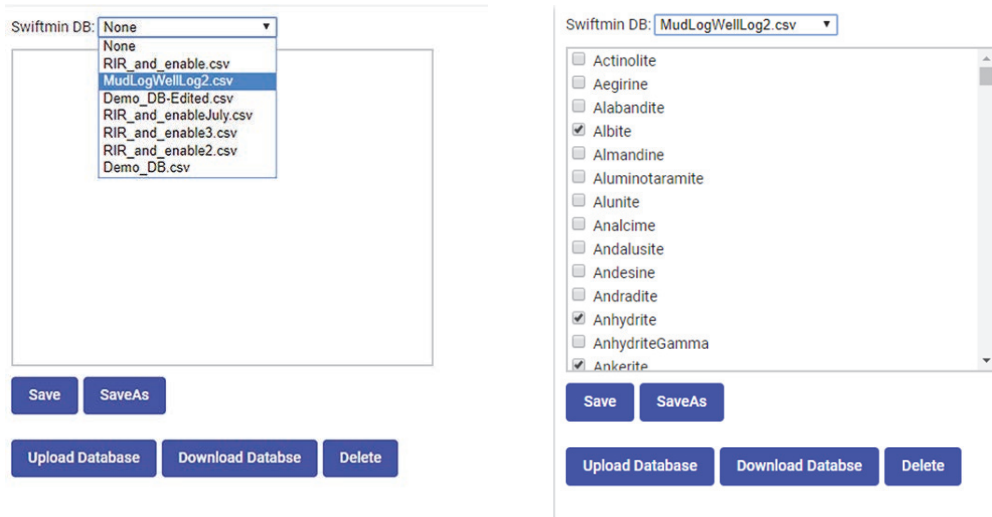


Figure 5-8 Choosing a database (left) and editing the mineral list (right)

To upload a .csv or .txt mineral database

1. Click **Upload Database** to open a file explorer window (see Figure 5-9 on page 75).
2. In the file explorer, navigate to your database file directory.
3. Select a database file, and click **Open**.

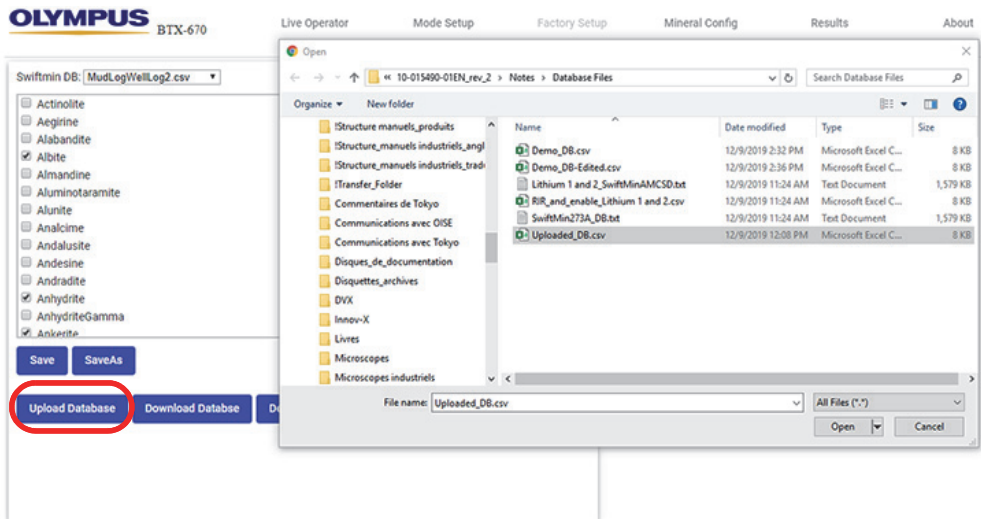


Figure 5-9 Upload database

To download a .csv or .txt mineral database

1. Click **Download Database**.
2. In the dialog box, click the down arrow beside the database you want to download (see Figure 5-10 on page 76).

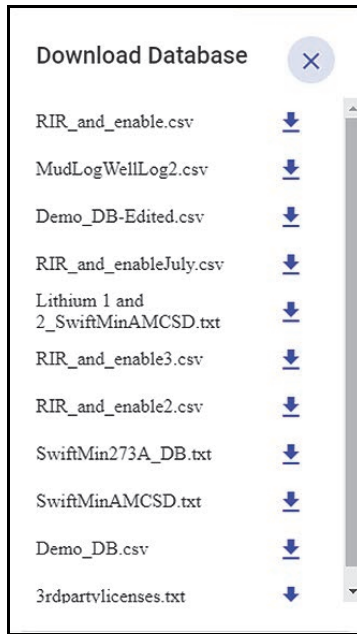


Figure 5-10 Download Database dialog box

To set the calibration

- ◆ Click **Set Calibration**.

The spectrum is selected to calibrate the SwiftMin® software for database searching, which ensures the identified materials are correct.

5.3.3.2 Mode Setup Tab

The Mode setup tab is where you set up the test modes for selection in the Live Operator screen. You can change default test mode parameters, or add, edit, or delete modes. You can also change the current default database to either the AMCSD or another RIR mineral database.

The parameters for the default mode are displayed on the left side of the screen. The available custom modes are displayed on the right.

To change the default database

1. Click on a default database and select a different database (see Figure 5-11 on page 77).
2. Click **Save**.

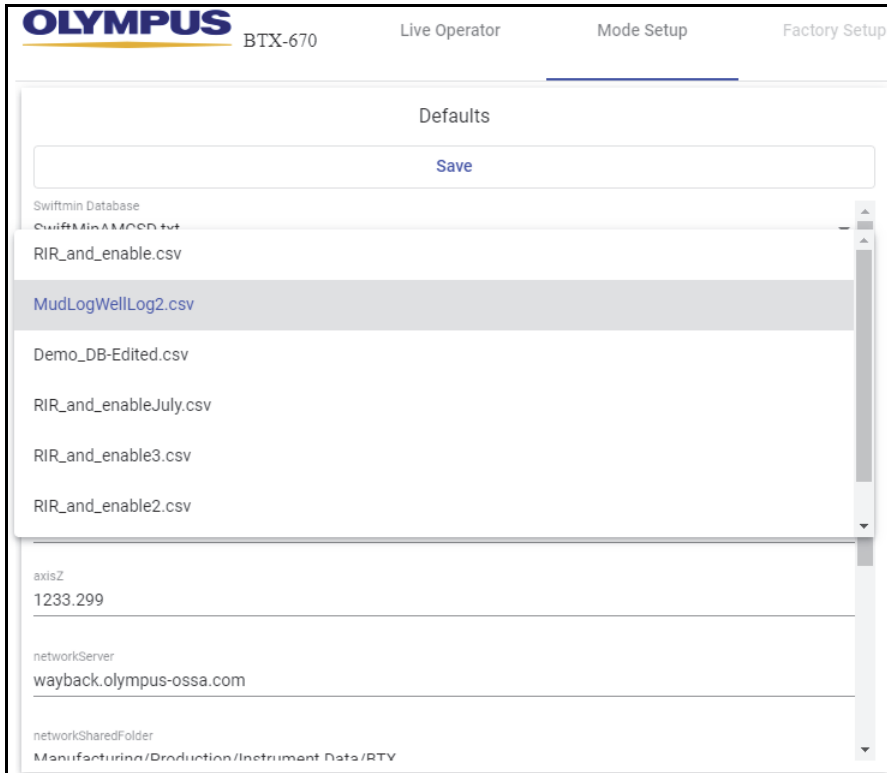


Figure 5-11 Changing the default database

To change any of the other default values

1. Highlight the value to select.
2. Type the new value.
3. Click **Save**.

To add a new mode

1. At the bottom of the **Custom Modes** dialog box, click **Add New Mode** (see Figure 5-12 on page 78) to display the **Add New Mode** dialog box.

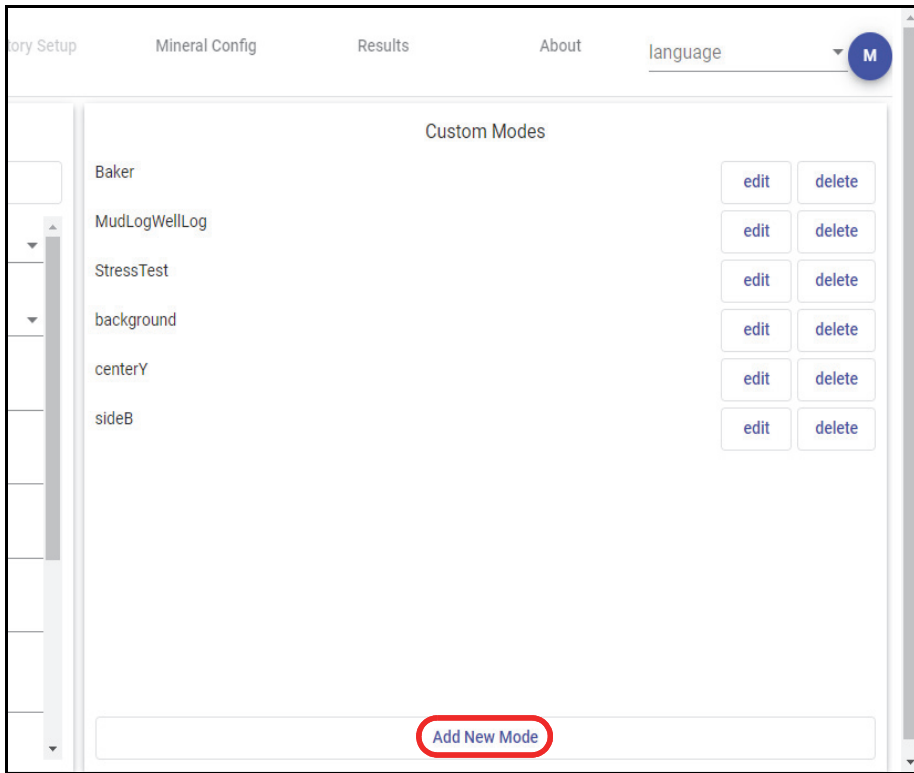


Figure 5-12 Add New Mode button

2. In the dialog box, select the **Add New Mode** field and type the name of the new mode (see Figure 5-13 on page 79).
3. Select the default database (see "To change the default database" on page 77).
4. Change any of the other default settings (see "To change any of the other default values" on page 77).
5. Click **Add**.

Add New Mode: NewMode

Default Settings Changed Settings

Swiftmin Database
SwiftMinAMCSD.txt

Swiftmin RIR
RIR_and_enable3.csv

exposures
200

multiRuns
1

piezoVolume
35

axisZ
1233.299

Cancel Add

Figure 5-13 Add New Mode dialog box

To edit an existing mode

1. Click an **Edit** button (see Figure 5-14 on page 80).

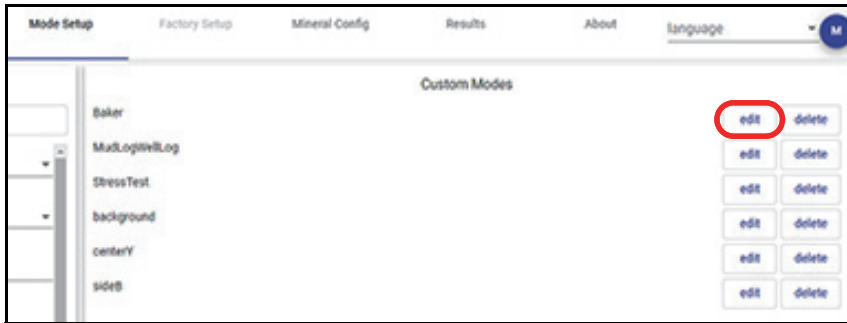


Figure 5-14 Edit buttons

2. In the **Editing** dialog box, (see Figure 5-15 on page 80) select the default database (see “To change the default database” on page 77).
3. Change any of the other default settings (see “To change any of the other default values” on page 77).
4. Optionally, select the mode name and then type to change the name of the mode.
5. Click **Save**.

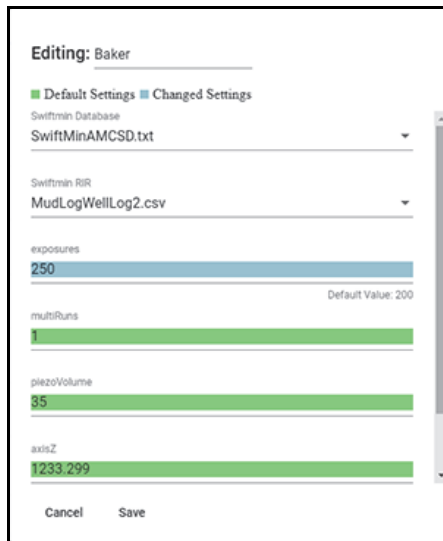


Figure 5-15 Editing dialog box

To delete a mode

- ◆ Click a **Delete** button (see Figure 5-16 on page 81).

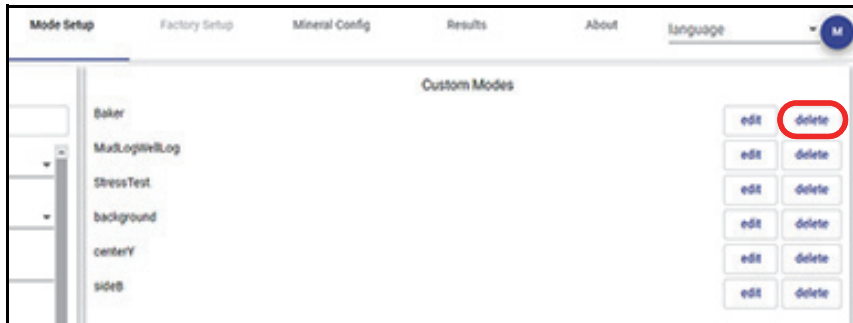


Figure 5-16 Delete buttons

5.3.4 Changing the User Interface Language

The **Language** tab enables you to change the language in the UI.

To change the user interface language

1. Click the language displayed in the **Language** tab.
2. Select a language from the list.

Appendix A: Specifications

The BTX III X-ray diffraction analyzer operating characteristics differ depending on the operating temperature. The BTX III analyzer consumes more power at higher ambient temperatures as it cools the CCD X-ray detector. See Table 9 on page 83 for full specifications.

Table 9 BTX III specifications

| Parameter | Specification |
|-----------------------|---|
| XRD resolution | 0.25° 2 θ FWHM |
| XRD range | 5–55° 2 θ |
| Detector type | 1024 × 256 pixels 2-D Peltier-cooled CCD |
| Sample grain size | <150 μm crushed powder (100 mesh screen, 150 μm) |
| Sample quantity | ~15 mg |
| X-ray target material | Cu (Co optional) |
| X-ray tube voltage | 30 kV |
| X-ray tube current | France: 360 μA Rest of the World: 330 μA |
| Data storage | 240 GB ruggedized internal hard drive |
| Wireless connectivity | 802.11b/g (enables remote control from web browser) |
| Operating temperature | -10 °C to 35 °C (14 °F to 95 °F) |
| Weight | 12.5 kg |
| Dimensions | 30 cm × 17 cm × 47 cm (11.8 in. × 6.9 in. × 18.5 in.) |

Appendix B: About Powder XRD Testing

The most commonly used crystallographic approach of XRD is powder X-ray diffraction (PXRD). The sample in PXRD is a powdered (polycrystalline) material, which is composed of many small crystallites that randomly assume all possible orientations with respect to the incident beam. In a PXRD experiment, a relatively small proportion of the grains contribute to a given diffracted beam. Higher numbers of randomly oriented grains exposed to the X-ray lead to better statistical representation for any given diffraction direction. This is referred to as *particle statistics*.

Powder X-ray diffraction instruments require limited analytical volume to provide good resolution, so particle statistics are achieved using very fine grains of typically smaller than a few tens of micrometers. Particle statistics become even more critical with miniature systems because of the reduced size of their analytical volume. Conditions for good particle statistics vary, depending on parameters such as the symmetries in the crystal lattice, the abundance of the phase in the sample, and the geometry of the system. A general rule of thumb for powder XRD is that at least 106 grains are needed to provide appropriate particle statistics. This is achieved in the BTX III with sub-micron powders that lead to continuous Debye rings when using static samples. Very spotty rings are observed for grain sizes above 10 μm (the term *pottiness* is often used to refer to insufficient particle statistics).

When the grain size of the sample is not small enough to guarantee appropriate particle statistics, means to increase the number of crystal orientations effectively analyzed must be applied. This is typically done by translating or rotating the sample in the beam to analyze a larger amount of material or explore more orientations of the same grains. A novel method employed in the BTX III XRD analyzer consists of placing the granular sample in motion using granular convection in vibrated cells. This method is very effective at improving particle statistics and enables analysis of materials with a grain size of up to 150 μm . This sample handling method relaxes the constraints on sample preparation by allowing grains nearly two orders of magnitude larger than ideal to be analyzed, while facilitating loading and removal of the powder.

See Figure B-1 on page 86. In example A (a still sample), partial diffraction rings and Laue spots are observed. In example B, with granular convection, complete diffraction rings are observed (what is known as a perfect powder pattern).

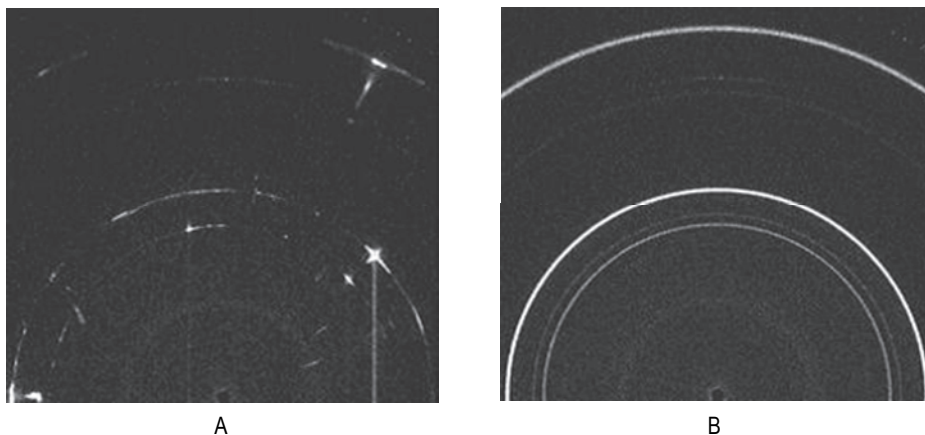


Figure B-1 BTX III XRD diffraction patterns: NaCl crushed and sieved to <math><150\ \mu\text{m}</math>

Appendix C: Data Analysis

The XPowder application software can be used to accomplish in-depth data analysis of a wide range of powdered substances. This software is supplied with your BTX III X-ray diffraction analyzer on the USB flash drive. This appendix includes instructions on installing the software and using it to verify alignment of the system using a quartz sample (not supplied).

To install XPowder


1. Connect the USB flash drive (supplied) to a USB port of a computer.
2. Find the **xpowder_setup.exe** () file, and then double-click the icon to run the program.
3. Follow the instructions in the setup wizard (see Figure C-1 on page 88) as the installation progresses.



Figure C-1 XPowder setup wizard

4. When the installation is complete, copy the registration code from the “License key” document on the USB flash drive.
5. Click **Main menu > Help > XPowder registration code** and paste the license key in the text box.

To load the DIFDATA database

1. Click **Database > Database Install**.
2. In the **Drive** list, click the down arrow to locate the USB flash drive (see Figure C-2 on page 89).
3. Under **Click database file**, select the **Difdata.txt** file.
4. In the **Database file extension** list, click the down arrow, and then select ***.txt**.
5. Under **Default scanning interval (Angstroms)**, in the **Higher d-spacing** box, type **70.00**, and then, in the **Lower d-spacing** box, type **1.64**.
6. In the **Database nickname** box, type **AMCSD**.
7. Select **Add a new Database**.
8. Click **Install**.

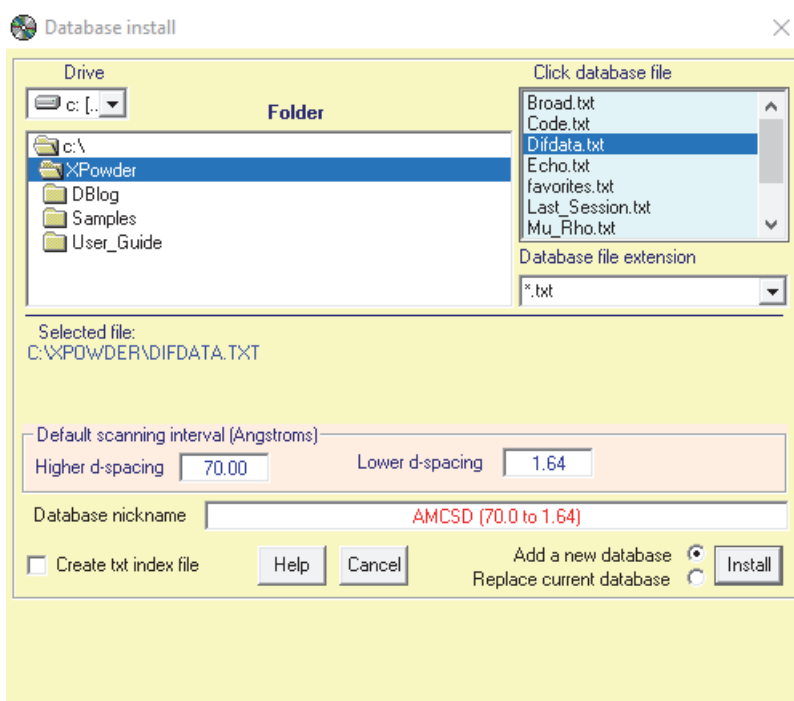


Figure C-2 Database install

To load test data

1. Click **File > Open**.
2. In the file type list change the file type to **x,y (ascii.txt, asc, xy,x_y)** [see Figure C-3 on page 90].

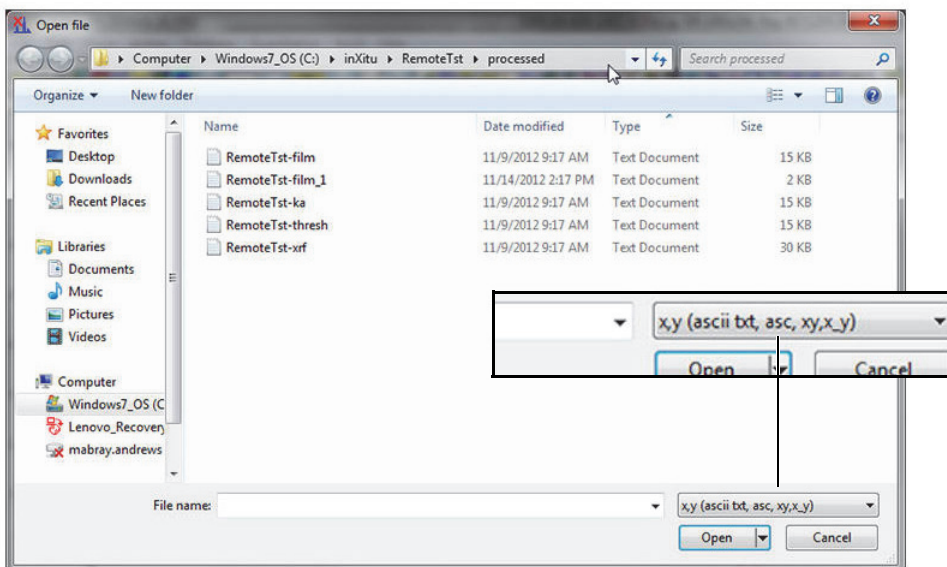


Figure C-3 Open File dialog box

3. Locate the file containing your quartz sample results. Ensure the file name ends with **-film.txt**
4. Click **Open** to display the XPowder home screen and the superimposed **Wavelength setup** dialog box (see Figure C-4 on page 91).

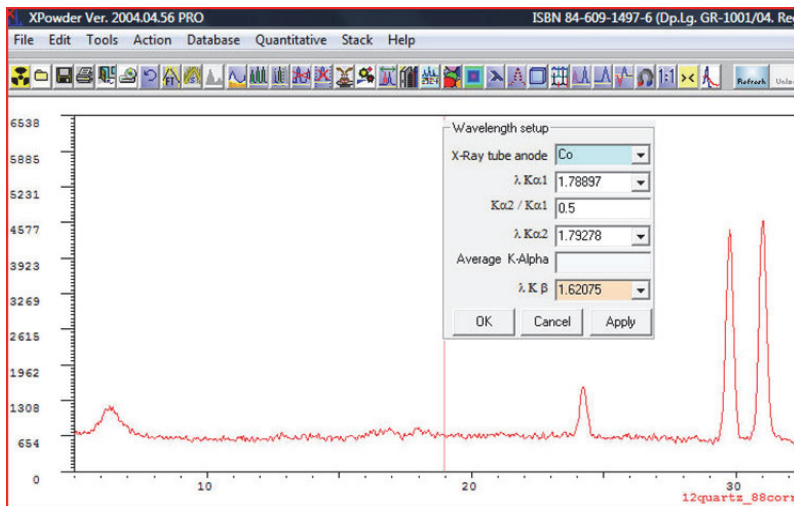


Figure C-4 Results screen and Wavelength setup dialog box

NOTE

The **Wavelength setup** dialog box is used to set the X-ray tube type, and automatically displays only on initial software setup.

5. In the **X-ray tube anode** list, select either **Co** or **Cu**.
6. The selection depends on the type of X-ray tube in your system.
7. Click **OK**.

To analyze the test data

1. In the **XPowder** menu, click **Action > Background subtraction** to display the **Background subtraction** dialog box (see Figure C-5 on page 92).

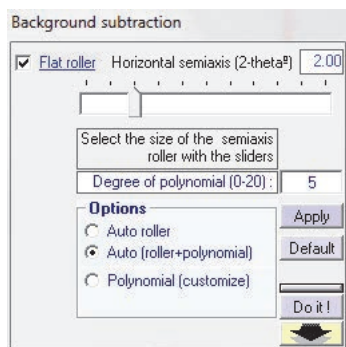

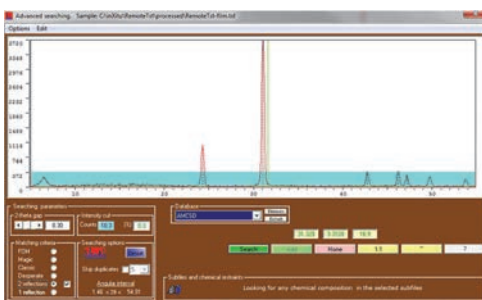
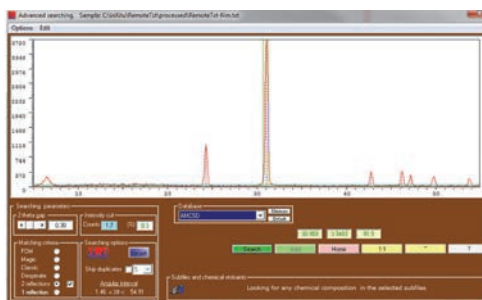


Figure C-5 Background subtraction menu

2. Without changing any of the parameters, click **Do it!**
3. Click **Yes** in the confirmation dialog box.
4. On the X Powder tool bar, click **Advanced searching** ()
The advanced searching window containing a diffractogram appears.
5. In the diffractogram, click within the (blue) background subtraction area to decrease the amount of background subtraction (see Figure C-6 on page 92).
The purpose of decreasing or increasing the amount of background subtraction is to eliminate as much noise as possible while preserving as many peaks as possible.



Background subtraction set too high — Some peaks are eliminated.



Background subtraction decreased — Most peaks are preserved.

Figure C-6 Advanced searching windows

- In the **Searching parameters** area, set the **2-theta gap** to **0.30** (see Figure C-7 on page 93).

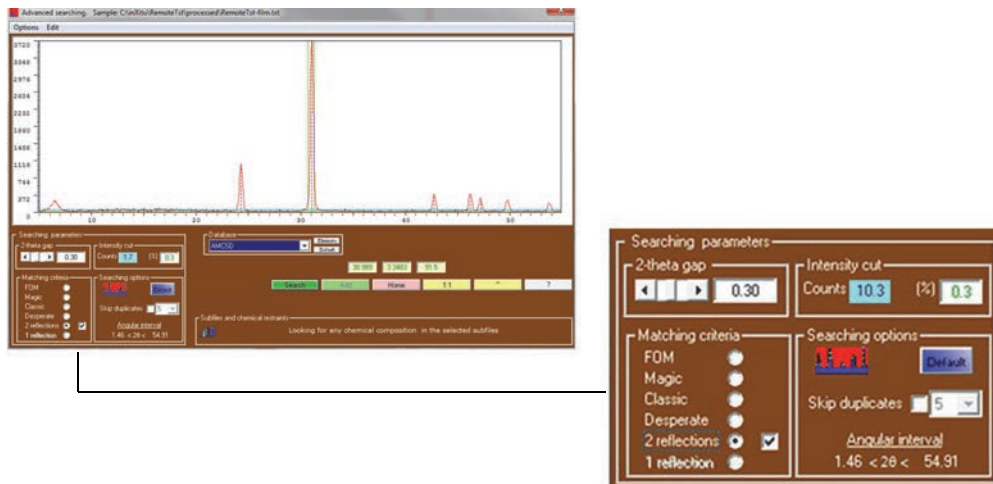


Figure C-7 Advanced searching — Searching parameters area

- In the **Matching criteria** box, select **2 reflections** option (both the radio button and check box).
- Click **Search**.

The search results are displayed in the XPowder window with a dialog box superimposed (see Figure C-8 on page 94). The dialog box name is based on the name of the CCD image file exported from the BTX II. In Figure C-8 on page 94, the dialog box name is **RemoteTst-film in AMCSD**.

XPowder window

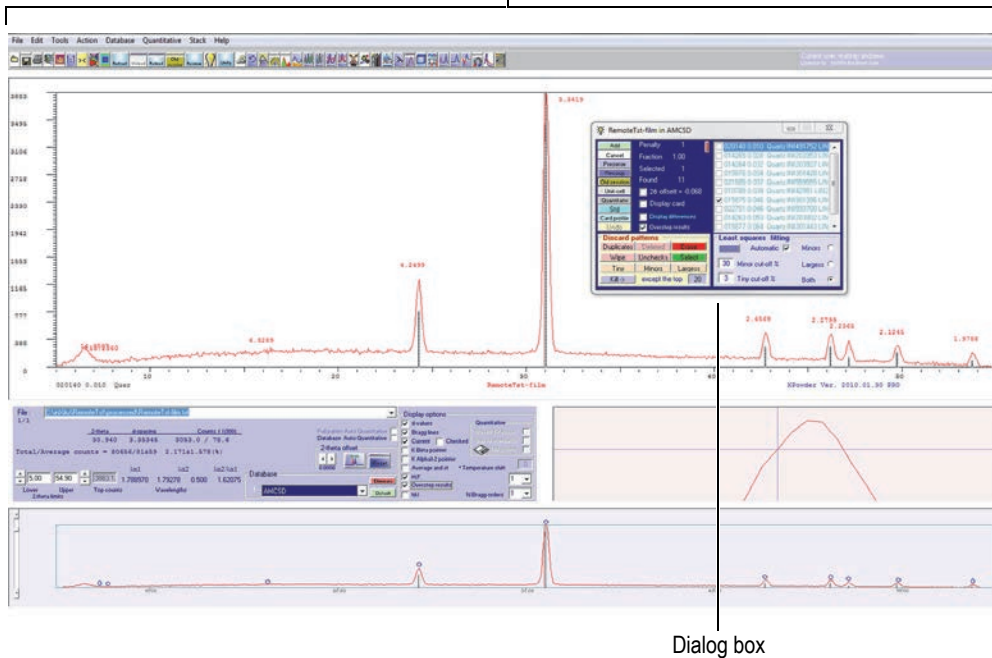


Figure C-8 XPowder results window

9. In the **Least squares fitting** area of the dialog box, click the **Automatic** check box. The XPowder software identifies the first crystal/phase that best matches the pattern.
10. In the phase list, click the check box of the highlighted phase to identify the next phase most likely to fit the pattern. In many cases, this is another isomer of the same crystal/phase.
11. Click **Unchecks** to eliminate unchecked (unwanted) phases (see Figure C-9 on page 95).

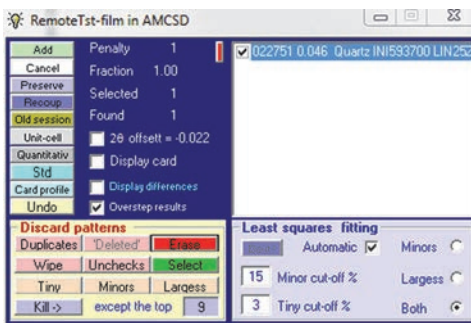


Figure C-9 Dialog box showing the selected phase

- Click **Display card** to inspect the phase details (see Figure C-10 on page 95).

| Name | Quartz | Std | Print | Info | | | | | | |
|----------------|----------------|-----------------|--------|---------------|---------|--------|-----------|----------|---------|--------|
| Chemical | SiO2 | | | | | | | | | |
| Set | 2 | File | 2751 | Subfiles | Mineral | SiO2 | X density | 1 | Abs RIR | 25.573 |
| Crystal system | Hexagonal axis | Space group | P3_121 | N# | 0 | a axis | 4.914 | Pres kPa | α | 90 |
| Anode | Co | Temp °C | 25.0 | b axis | 4.914 | β | 90 | | | |
| Lambda | 1.78897 | Pattern quality | C | Active record | | c axis | 5.406 | γ | 120 | |

Figure C-10 Phase detail display card

- In the X Powder window, click **Quantitative > LS-RIR Database Cards** to view a relative intensity ratio semiquantitative analysis and summary (see Figure C-11 on page 96).

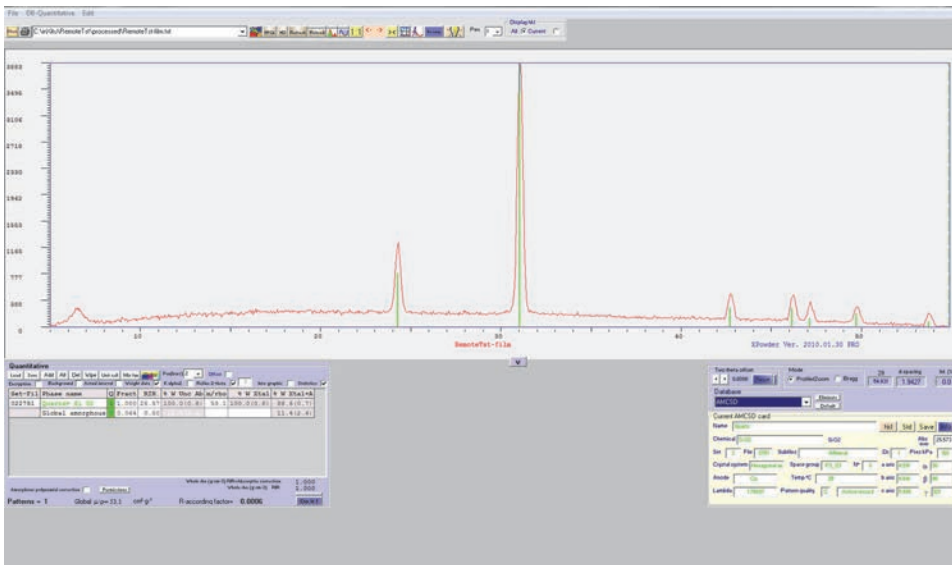


Figure C-11 Quantitative screen

14. To view a log report, click **File > Save log file as TXT** to generate a text file record of the session.

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