NIRS
XDS SmartProbe Analyzer

Manual
8.921.8004EN

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Although all the information given in this documentation has been checked with great care, errors cannot be entirely excluded. Should you notice any mistakes please send us your comments using the address given above.

Change Control

Version	Date	Description of Changes
1.04	March 03, 2005	Add Change Control section Updated company address to reflect move to new location
1.05	July 27, 2009	Updated headers and Table of Contents to current style. Updated Network Connection to current recommendations. Replaced photos showing XC-1200 Standard on probe, clarified Reference Standardization explanation. Updated various menus to current XDS style. Moved Wavelength Linearization before Reference Standardization. Replaced section on XDS Instrument Connection with an updated, more current version. This also includes basic connection troubleshooting. The sections on Windows 95, 98, and NT 4.0 were dropped. Updated explanations for Instrument Calibration and Wavelength Certification. Added photos to some procedures to clarify required probe positions. Added note that USP dropped "Photometric Linearity Test" as of 12-2008. Added notes about possible failure of Photometric Test, and corrective action. Corrected wrong instruction in Gain Test. Reflectance probe is normally used in Gain. Changed photo in Lamp Replacement, now showing arrow on lamp. Corrected reference to Lamp Replacement in Troubleshooting to section 8.3. Updated Vision "opening screen", removed copyright date for publication. Updated Configure, Options screen and discussion to current version. Added note to Troubleshooting, Instrument Calibration, that Reference Standardization must be performed. Added note about wavelength drift due to temperature change with the WSR standard. This is on the page immediately following Instrument Calibration. Added note about 21 CFR Part 11 Verification document to Validation section. Added note to avoid scratching sample probe window to maintenance section. Added troubleshooting items which may be symptomatic of network connection issues. Added note on page in section on Interactance Reflectance, to avoid carrying dirt or sample material from the Smart Probe barrel to the instrument reference.
1.06	February 21, 2012	Corrected description of instrument reference in Wavelength Linearization.

Table of contents

1 Introduction

Thank you for selecting the XDS™ Smart Probe Analyzer, manufactured by FOSS. This instrument is the third generation in a series of instruments designed for precision NIR measurement, characterization of organic materials, and qualification of known materials to quality parameters.

The XDS™ is designed for stable operation in harsh environments, while still providing the precision and accuracy users have come to expect of FOSS instruments.

Standard fiber bundle lengths are 2-meter and 3-meter. The 3-meter fiber bundle is shown here.

The Smart Probe Analyzer uses a proven monochromator design, employing a digitally-controlled dispersive grating, along with sensitive detection devices and state-of-the-art circuitry to enhance signal output and minimize any extraneous noise that might influence performance. The XDS™ uses various patented algorithms to provide superior accuracy and transferability between like instruments.

This instrument uses near-infrared (NIR) spectral energy to illuminate the sample. By measuring the energy reflected off (or passing through) the sample, chemical information and composition may be determined. This information may be used for quantification of constituents, or for comparison to a library of known materials, providing identification and qualification of materials.

The Smart Probe Analyzer has been a versatile, proven design for NIR sampling for many years. As an XDS™ instrument, the Smart Probe benefits from the improvements in wavelength accuracy, precision, low noise, speed, and overall performance.

A panel of 6 LED indicators provides information to the user on these functions:

Icon	Status
	Green when power is ON
	Amber when connected to network or direct connection
	Green when instrument lamp is ON
	Red when scanning reference or sample

Green when stable operating temperature is reached

Green when module is properly attached

Vision Software™ offers an easy user interface, using the familiar interface provided with previous generations of NIR instrumentation. All functions required to perform identification, qualification, and quantitation are provided, with easy tools for interpretation of results. Vision offers full instrument diagnostics, with built-in acceptance specification tables for all tests. Vision stores all results in a diagnostic database for later lookup, with control chart views of results tracked over time.

The menu-driven, validated Vision Software package meets all requirements of CFR 21 Part 11, covering Electronic Records and Signatures. Vision comes with a full manual for operation and theory of operation, with complete instructions for analytical development.

The XDS Analyzer provides 0.5nm data points, and uses several innovative methods to assure wavelength accuracy and repeatability. Wavelength positions are traceable to NIST SRM-1920a. Because SRM-1920a does not have certified wavelengths above 2000 nm, an additional wavelength absorber is included in the calibration standard, to provide stable wavelengths beyond 2000 nm. These additional wavelengths have been independently measured on calibrated instrumentation to ascertain the wavelength positions used.

Instrument communication is through RJ-45 network connections, which eliminates issues involved with long runs of RS-232 cable. An Internet Protocol (IP) address is dynamically requested upon connection. This address may be permanently installed, if required for network purposes. The RJ-45 connection also permits remote interrogation and diagnostics checks of the instrument, if necessary and authorized.

The instrument enclosure is completely sealed to prevent contamination by dust or other substances. The cooling fans operate outside the main enclosure, and are thermally linked to internal fans that maintain a constant temperature inside the instrument enclosure. There is no airflow drawn into the optics chamber instrument. An external fan-cooling loop is provided in the side chassis, with thermal conduction from the inside of the optics chamber. This avoids contamination of the instrument in dusty environments. An air filter is built into the door of this chamber. For cool environments, heaters are embedded in the thermal transfer block to raise temperature when required.

Lamp changes are performed through a single panel on the rear surface of the instrument. The lamp is easy to remove and replace, and requires no special tools or expertise.

LED Indicators on the Smart Probe Handle

Pass:
The sampled material has passed the ID and Qualification test (Routine Analysis)

Fail:
The sampled material has failed the ID or qualification Test (Routine Analysis) This means the material was either not identified, or was outside of acceptance limits for that material.

Ready:
The Smart Probe is ready to take a sample scan.

Scan:
Scanning reference or sample.

Enter:
Not used

Ref:
System is ready to scan reference material

2 Site Readiness

Like most precision instruments, the Smart Probe Analyzer is sensitive to environmental conditions that can affect its performance and useful life. Observe the following guidelines when selecting a site and installing the instrument:

2.1 Temperature and Humidity

The XDS Analyzer is designed to work in ambient air temperatures from 40-95°F (4.5-35°C).

Use the XDS Analyzer only in 10-90% relative humidity levels, non-condensing. Rapid changes in humidity can cause interferences by adding trace moisture absorptions to the spectra. In general, lower humidity levels are preferred.

The Performance Test (a comprehensive instrument diagnostic test in Vision software) is somewhat sensitive to changes in ambient humidity, and the Performance Test may fail under conditions of extreme humidity, or rapidly varying humidity.

2.2 General Environment

Minimize exposure of the monochromator to dust.

Inspect the fan filter at least monthly. If an accumulation of lint, dust, or other matter has accumulated, pull open the right-hand panel from the instrument. Replace the filter. If dust has accumulated on the fans, carefully wipe them clean with a moist soft cloth. Do not distort or damage the fan blades or fins, as this will impede cooling.

Do not place the instrument directly near any HVAC duct. The direct flow of heating or cooling air will cause the instrument to exhibit high noise during the Performance Test.

2.3 Electrical Power

Power should be a single, separate, stable, transient-free filtered AC circuit. The circuit should have surge protection.

Operating voltage for the instrument is 100-240VAC, 50/60Hz. The power supply is self-switching and will provide the correct operating voltage to the instrument.

Maximum power consumption is 750W.

2.4 Vibration

Install the XDS Analyzer where it will not be affected by bench vibration from grinders, blenders, stirrers, or mixers.

Never permit hammering or other physical impact on the bench top supporting the XDS Analyzer or its computer.

2.5 Instrument Communication

The XDS Smart Probe Analyzer can communicate directly with the computer by use of a UTP Crossover Cable, such as CDW #243786, supplied with unit. (gray cable)

Alternatively, the instrument may be accessed directly through a network connection. This uses a

standard RJ-45 type cable, such as CDW #074092, available from CDW Computer Centers, Inc. The instrument detects network capability and optimizes communication speed.

The computer that operates the instrument must have clear access through the network, and be configured to communicate properly. This communication is the responsibility of your on-site network personnel.

Full instructions are given in section 3.0.

2.6 Instrument Dimensions and Weight

XDS™ Smart Probe Analyzer dimensions are:

- **Width**: 18.0" (457 mm)
  (Leave about 6" (152mm) on the right side for access to standards door)
- **Height**: 15.0" (381 mm)
  Height with Probe: 17.5" (445mm)
- **Depth**: 22.5" (572 mm) front to back
  Depth with Probe: 32" (812mm)

Leave a minimum of 3" (76mm) around the back and left side for airflow and access space. Leave at least 6" (152mm) on the right side, and in front for fiber bundle space.

Weight: 67.2 pounds (30.6 kg)

Follow lifting instructions (on last page) when moving the instrument. Avoid injury.

3 Instrument Connection

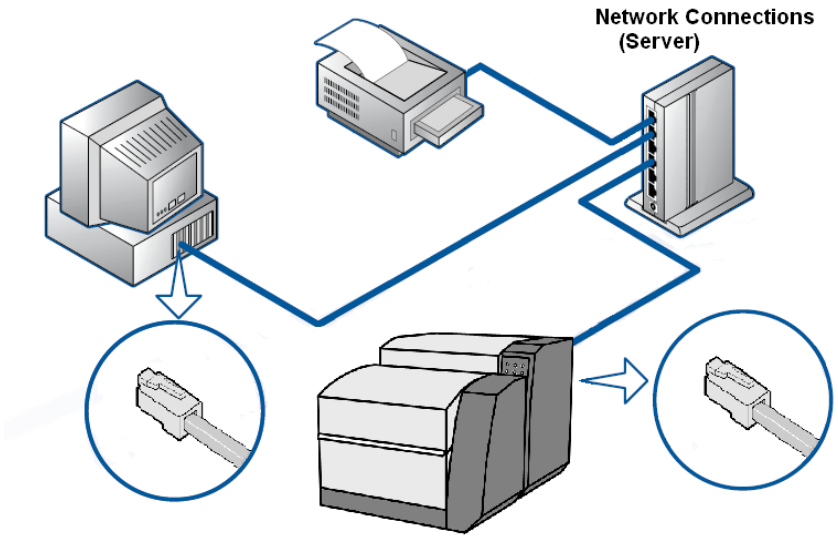
The XDS instrument may be connected to the host computer in one of two ways: If the XDS Instrument will be used as part of a network, use the Network Connection method shown immediately below. If there are not enough active network ports near the XDS instrument, a hub or router may be used.

This section assumes use of Windows® 2000, XP, or later versions of Windows Operating Systems. For computers using Windows 95, 98, or NT 4.0, we recommend upgrade of the computer and operating system to current specifications.

CAUTION: Metrohm NIRSystems does not recommend the use of two network cards under any circumstances. Do not use Direct Connection to the instrument along with a network connection to the company network. The use of two network cards -- on one data bus in the computer – may result in lost commands, lost data, and unsatisfactory software operation. Metrohm cannot be responsible for software and instrument problems resulting from the use of two network cards in the host computer.

This information is correct as of the time of original publication. Changes to computers, operating systems, and network protocols may require revision of this information without notice.

3.1 Network Connection, connected to an active network port as shown

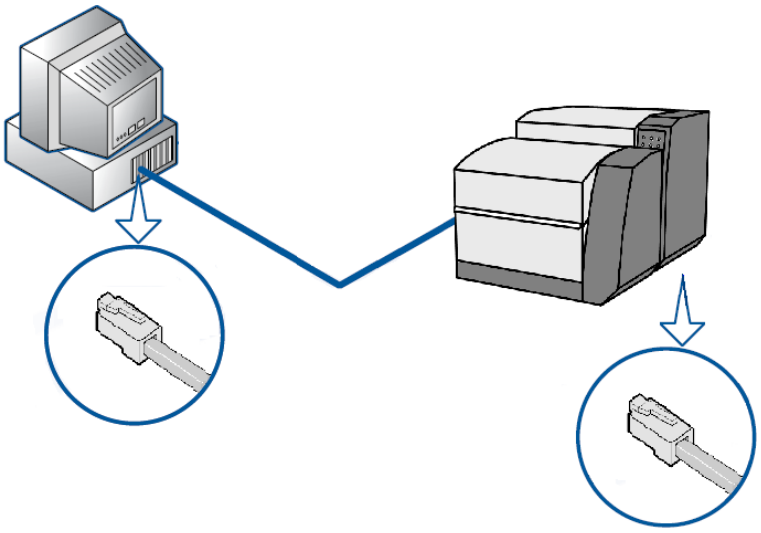


This is the preferred method of instrument communication when a connection to the company Local Area Network (LAN) is necessary. Specific information about this method follows:

- The XDS instrument should be connected -- with a "patch" cable – to the network port.
- Upon power-up, the XDS instrument will request a dynamic IP address from the network server. This is normally assigned in 5 to 10 seconds.
- The XDS instrument uses a proprietary, encrypted command language. It cannot be activated by any program except Vision, or FOSS programs designed to operate the instrument. Therefore, the instrument maintains "Closed System" status under 21 CFR Part 11 rules. No hacking or support of viruses is possible with XDS instruments.

- The XDS instrument appears just like a network printer (or other peripheral device) on the LAN system. It generates no signals, and only responds when commanded by an authorized user, logged into Vision software.
- This is the easiest connection method for XDS instruments.

3.2 Direction Connection, free-standing manner with no network connection



This method allows users to connect to the instrument when there is no network present. In such cases, a "crossover cable" (provided) is used. The XDS instrument, upon power-up, requests a dynamic IP address. When none is supplied within 45 seconds, the XDS instrument concludes that no DHCP server is available. It then defaults to an internal IP address which the computer may use for "direct communication".

This method of hookup should not be used when the computer is also connected to a network. Such connection may result in lost commands, lost data, and unsatisfactory software operation. FOSS cannot be responsible for software and instrument problems resulting from the use of two network cards in the host computer.

For IT personnel, it may be helpful to understand the sequence of events used by the XDS instrument and Vision software when establishing an electronic connection. These are explained.

3.2.1 Overview of XDS Instrument Communication

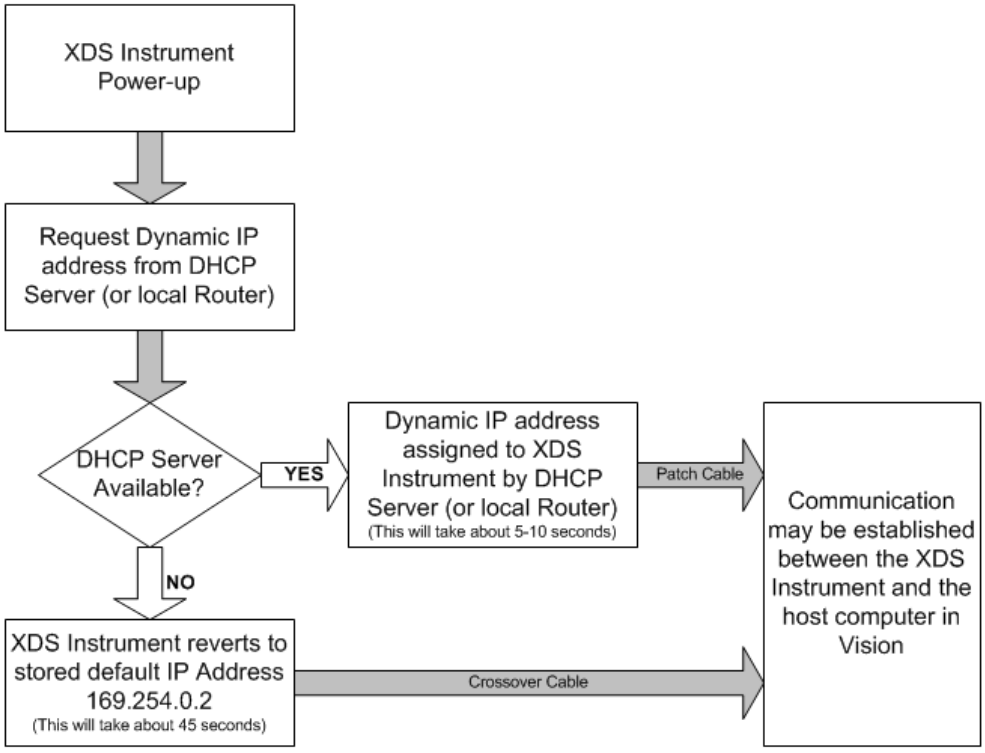
The XDS instrument may be connected to LAN systems in the same manner as any printer or other peripheral Ethernet-enabled device. These key items will help understand the communication methods. See the flowchart diagram on next page.

1. The XDS instrument maintains "Closed System Status" under 21 CFR Part 11 guidelines. It uses a proprietary, encrypted command language. It is not susceptible to hacking or virus attacks.
2. The XDS system may only be addressed using proprietary software (usually "Vision") which can only be entered by an authorized user, using the "two-token" method of entry. (Unique User ID and password)

3. Upon being powered up on a LAN, the XDS instrument requests a "dynamic" IP address from the DHCP server which controls the LAN. This IP address is normally granted promptly (typically in 5-10 seconds) so the instrument can function on the LAN. Most DHCP servers track the XDS instrument by the "MAC" (Machine Access Code) to later re-assign that same IP address whenever the XDS instrument is on the LAN.
4. If there is no DHCP server available to assign an IP address (a free-standing router may serve the same DHCP function), the XDS instrument will "time out" in 45 seconds --and it will know that it is not attached to an active LAN. It will then default to an internally-stored default IP address. This address, 169.254.0.2, is used for local, free-standing communication only. In such cases, a crossover cable, or a hub with two patch cables, should be used to connect the computer and the XDS instrument.
5. Upon the next power-down and subsequent power-up of the XDS instrument, it will again request an IP address of the DHCP server. It will go through the same cycle, eventually reverting to the stored default IP address. This is intentional.
6. A dynamic IP address is the preferred method of XDS instrument connection. The default IP address is only used when no DHCP server is available to assign a dynamic IP address.

A short glossary of terms follows. See the flowchart diagram for XDS instrument communication which visually outlines the items explained above.

Flowchart Diagram of XDS communication protocol



3.2.2 Microsoft Windows Firewalls

The Microsoft Windows® Firewall on the PC may interfere with Vision communication. To assure communication, follow these steps:

- Enter Control Panel, Security Center.
- On the "General" Tab, be sure that "exceptions" are allowed. (Un-click "Don't allow exceptions".)
- On the "Exceptions" Tab, click "Add Program".
- Select Vision from the list of programs – click on it. (Vision must be installed to appear on the list.)
- Click on "OK" at each window to exit Control Panel.

3.2.3 Network Evolution Issues

This document is as correct as possible at the time or writing. However, network management is an evolving discipline, and conditions will change. Some of the drivers for change include network security, authentication, and data integrity. Technology changes factor into all of these issues.

Because the network communication environment is complex and ever-changing, we have tried to provide the basic information needed for connection of the XDS instrument. 95% of users will have no connection problems, if these instructions are followed.

In the rest of the cases, there may be network issues, corporate restrictions, or other issues which inhibit easy connection. The troubleshooting section covers some of the most common problems.

In all cases, we recommend minimal tampering with computer settings. This can cause instability, and may be prohibited by company policies.

At this time, we recommend Microsoft Windows® XP as the easiest operating system by which to establish network communication. We strongly recommend that Windows 95, 98, and NT 4.0 be avoided, as they require considerable expertise in network configuration.

3.2.4 Quick Glossary of Terms:

DHCP:

The Dynamic Host Configuration Protocol (DHCP) is an Internet protocol for automating the configuration of computers that use TCP/IP.

DNS Server:

A Domain Name Server. DNS Servers run special-purpose software, as part of the Domain Name System, for managing enterprise networks.

IP:

An Internet Protocol (IP) address is a numerical identification and logical address that is assigned to devices participating in a computer network utilizing the Internet Protocol for communication between its nodes.

IPv4:

IPv4 refers to "Internet Protocol version 4" which is the fourth revision in the development of the Internet Protocol (IP) and it is the first version of the protocol to be widely deployed. Together with IPv6, it is at the core of standards-based internetworking methods of the Internet and is still by far the most widely deployed Internet Layer protocol. XDS Instruments use IPv4.

LAN:

A local area network (LAN) is a computer network covering a small physical area, like a home, office, or small group of buildings, such as a corporate site, a university, or an airport. These are often called "enterprises".

Subnet Mask:

"Subnetting" is used to break a large network into smaller sections. This can enhance efficiency, raise speeds, and reduce "packet collisions" within the network. To accomplish subnetting, "Subnet Masks" may be applied to separate one section of the network from another. A subnet mask typically takes the form "255.255.255.0" or something similar. This scheme is becoming obsolete, as new network management methods are being implemented.

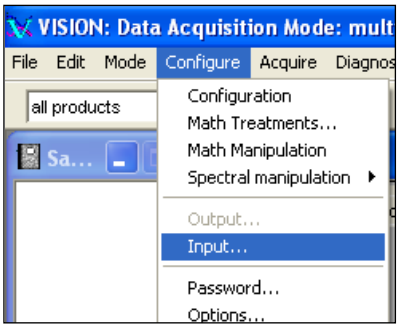
TCP/IP:

(Transmission Control Protocol/Internet Protocol) is the basic communication language or protocol of the Internet.

3.2.5 Connection in Vision

1. Log into Vision with your User ID and Password.

   Click on Configure, Input as shown.

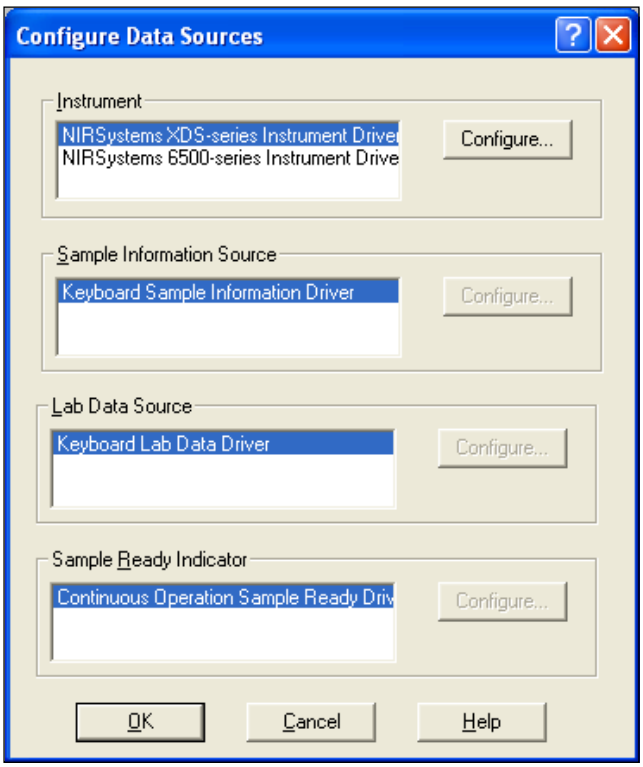


2. Highlight "NIRSystems XDS-series Instrument Driver" as shown, then click on "Configure".

   Information:

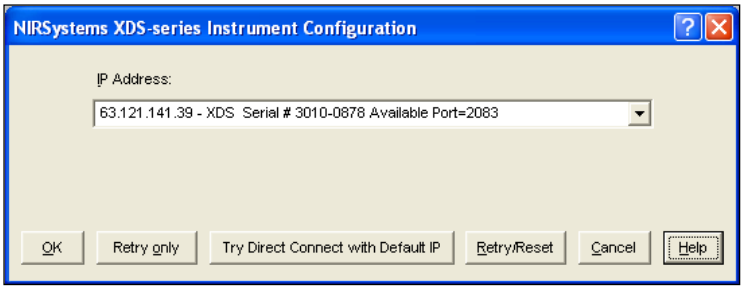
   At this point, Vision requests any XDS instrument on the local area network (LAN) to report connection status. This may take a few moments.

   If the instrument is not on a LAN, and instead is connected with a crossover cable, this will take a minute or more. Vision first requests a dynamic IP address, If no server or router is available to assign an IP address, Vision waits 45 seconds, then searches for the default instrument IP address, in the event of Direct Connection using a crossover cable.



3. When Vision "finds" the instrument on the LAN, it will be shown. The dynamic IP address (assigned by the server) is shown, along with the XDS Serial number. The instrument is shown as "Available" on port 2083.

   Highlight the instrument and click "OK".



4. 4. If the IP Address field is empty, the user should consult "Troubleshooting Connection Problems".

3.2.6 Troubleshooting Connection Problems

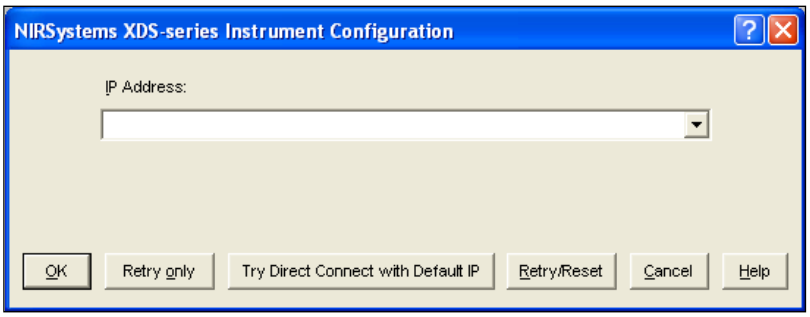
Many connection problems are easily solved, especially with Windows® XP operating systems. Windows XP is currently the preferred operating system, and has enhanced connectivity over other operating systems. Vista is good also, but may impose user security restrictions. Windows 2000 is almost as simple, but may require an extra step or two, as discussed.

If your computer uses Windows 95, 98, or NT 4.0 for the operating system, we strongly recommend upgrade to Windows XP for easiest connectivity. This may require a full computer upgrade, as older

computers may not have the processor speed, memory, or connectivity required to run Windows XP with full Ethernet compatibility.

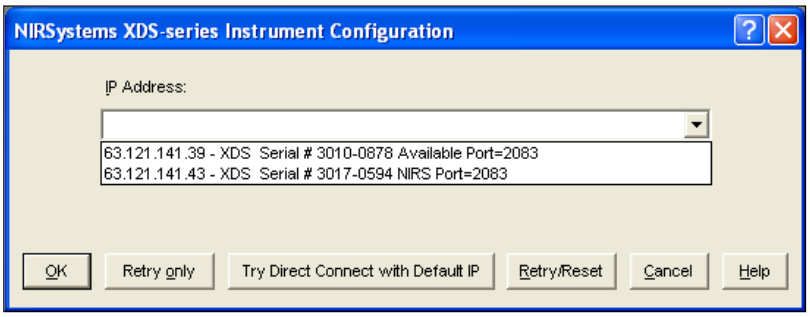
1. 1. Vision cannot see any instrument on the connection path.

   Click on the "down arrow" at the right side of the empty field to see if instrument(s) are shown.



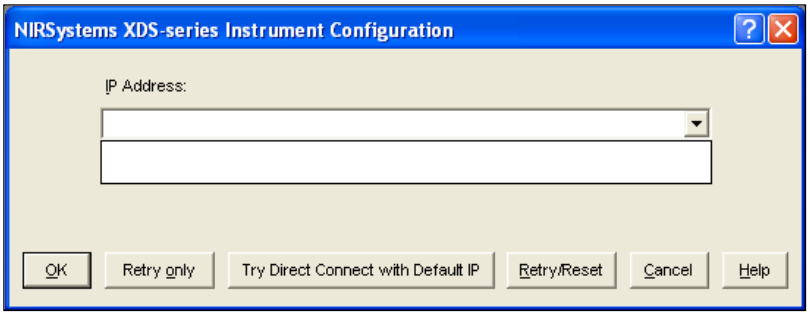
   Solution: By expanding the field, Vision can display the instruments shown.

   Note that only the top instrument, Serial #3010-0878, is "Available". Highlight it and click OK".



2. Vision still sees no instrument(s) after expanding the field.

   This indicates connection or network issues.



Verify Cable Type:

Verify correct cable type for hookup. Most networks use "patch" cables. Free-standing systems use a "crossover cable". Power down the XDS instrument, then power it back up. Wait 120 seconds for the XDS instrument to fully reset its communication. If an instrument is shown, proceed to "Acquire", "Connect" in Vision.

If this does not resolve the problem, continue to the next section.

3.2.7 Network Troubleshooting Overview:

If no XDS instrument shows as "available", there may be a setting which should be changed. It may be necessary to contact your IT department for assistance with these issues.

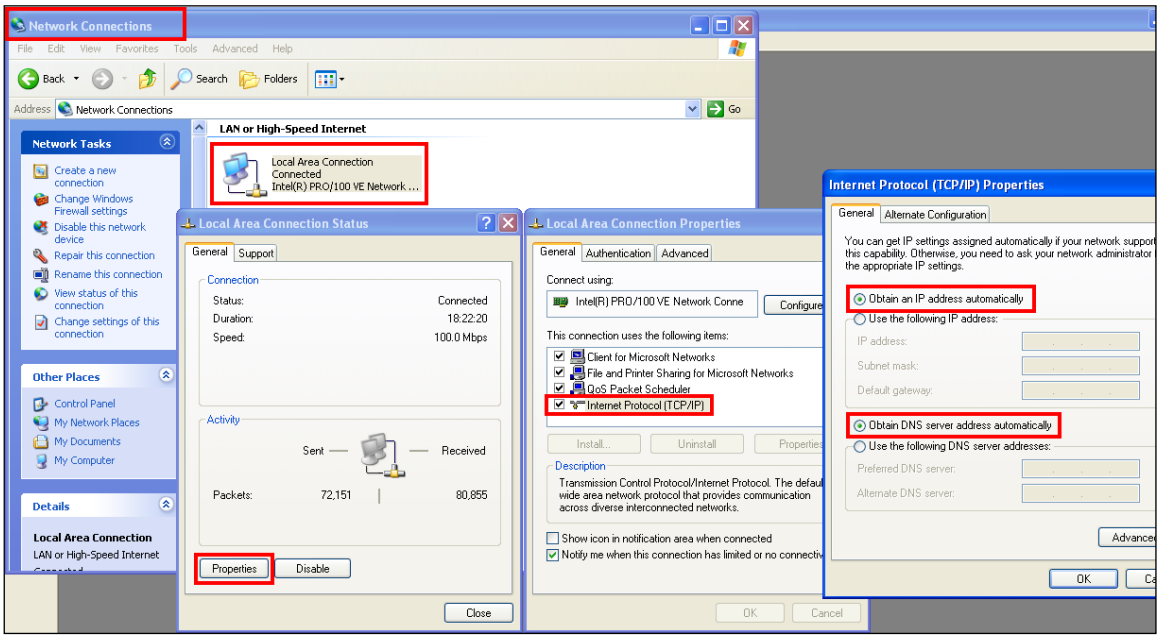
First, verify that the network has a DHCP Server. If no DHCP server is available, the instrument must be connected by Direct connection, using a crossover cable. If this is the case, proceed to the section entitled "Direct Connection Troubleshooting Overview".

Network Solution 1:

Check Internet Protocol (TCP/IP) Properties. (You may need to contact your IT department to follow these steps.)

- Click on Start, then Control Panel
- Double-click on Network Connections
- Double-click on Local Area Connection
- Click on Local Area Connection Properties
- Click on Internet Protocol (TCP/IP)
- Click on Properties

The full path, from Network Properties forward to Internet Protocol (TCP/IP) Properties, is shown:

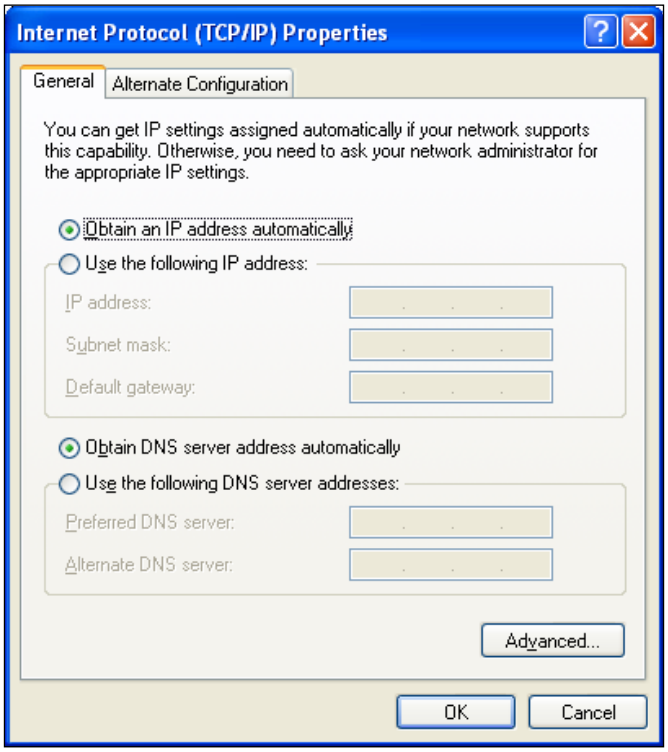


Verify these settings:

- Obtain an IP address automatically
- Obtain DNS server address automatically

When finished, click "OK". Close all other boxes opened for this verification.

If the settings were not set properly, it may be necessary to exit Windows XP, then re-enter XP, to have the correct settings take effect. If in doubt, do this and try XDS instrument communications again after this takes effect.

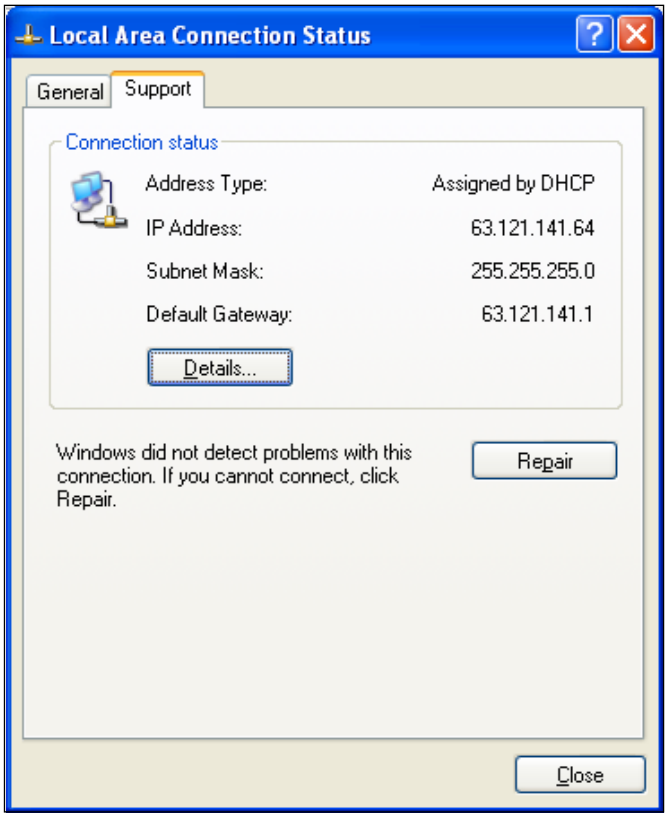


Network Solution 2:

Returning to the Local Area Connection Status dialog box, note these items for the computer:

- Address Type:
  (should be "assigned by DHCP")
- IP Address:
  Write this address down for the next step
- Subnet Mask:
  Write this down for the next step

If connection cannot be achieved, it may be necessary to verify that the XDS instrument is installed "within the IP address range" of the computer.



Network Solution 3:

Verify network has full IPv4 compatibility.

Some networks have moved to Ipv6 (Internet Protocol version 6) which uses different address formats.

The IT department at your company can verify if the network offers full Ipv4 compatibility. If the network has migrated to Ipv6 operation, a "compatibility pack" may need to be loaded to support Ipv4-enabled devices.

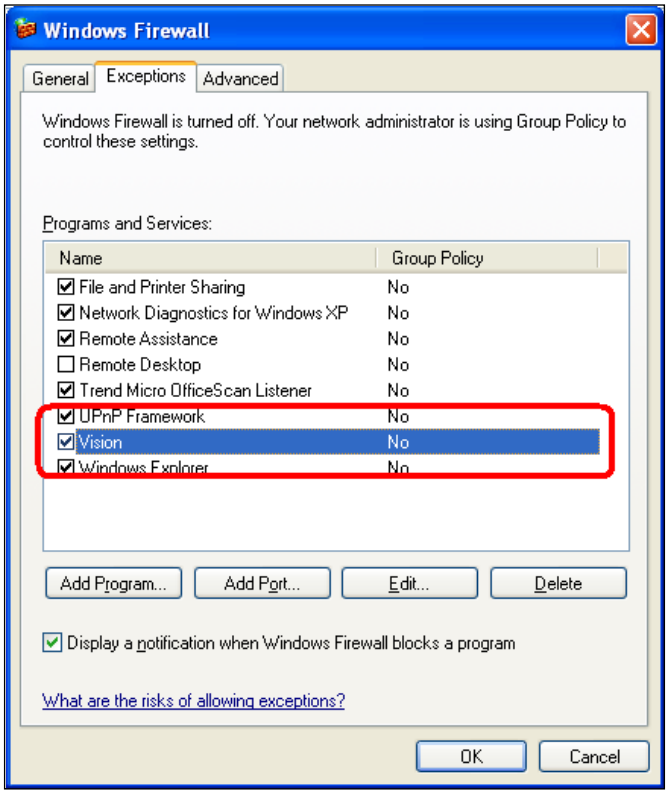
Network Solution 4:

Verify that the Firewall on the computer has Vision loaded as an "exception".

This is located under "Control Panel", "Windows Firewall".

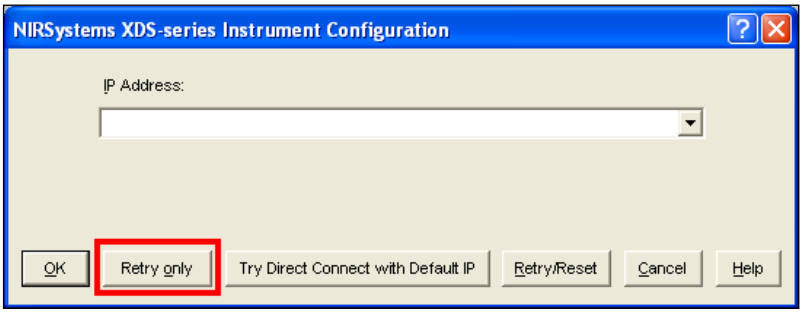
If this is not enabled, click "Add Program" and select "Vision" from the list.

When finished, click "OK"



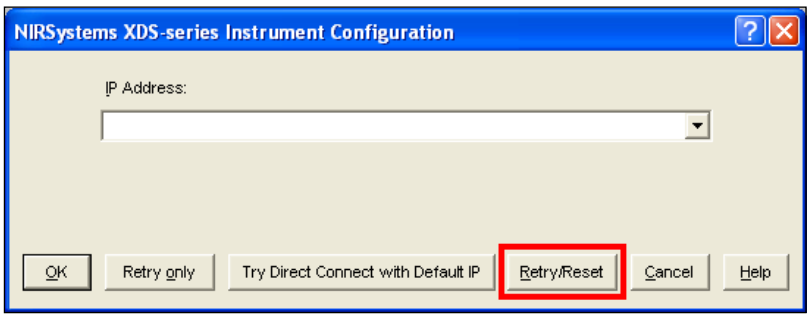
Network Retry, XP and Vista:

Windows XP and Vista users should click on "Retry Only". This command resets the communication port, and allows Vision to "find" the instrument, if connected properly.



Network Retry, Windows 2000:

Windows 2000 users should click on "Retry/Reset". This command resets the communication port, and also resets Windows 2000 to the proper state to connect using a dynamic IP address in the XDS instrument.



3.2.8 Direct Connection Troubleshooting Overview:

If no XDS instrument shows as "available", the computer may need to be configured for the IP address range of the XDS instrument. It may be necessary to contact your IT department for assistance with these issues.

First, verify the following:

- The instrument is free-standing not connected to a network with DHCP server
- There is only one network card in the computer
- A crossover cable is used between the XDS instrument and the computer

If these conditions are met, please proceed.

Direct Connection Solution 1:

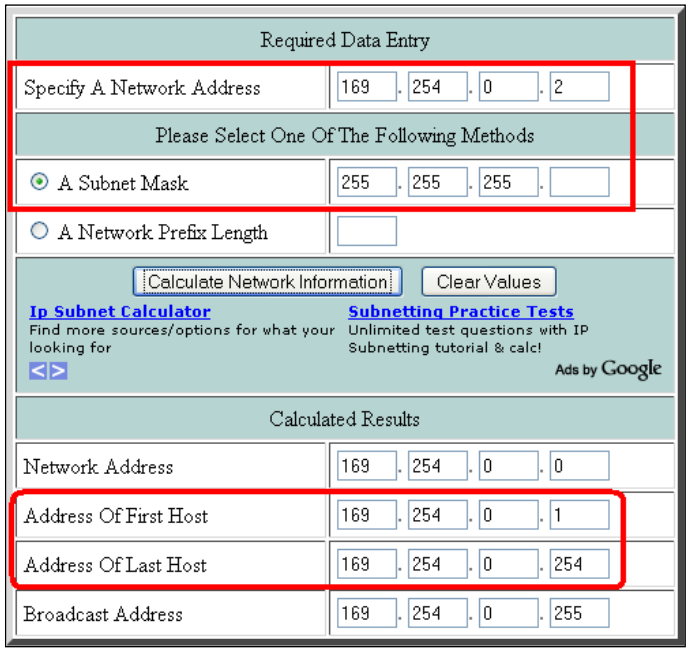
If using a crossover cable, verify that the computer is communicating in the same IP range as the XDS instrument.

The XDS instrument default IP address is 169.254.0.2, as shown. This address calculator gives the allowable computer IP address range as 169.254.0.1 through 169.254.2.254.

DO NOT use 169.254.0.2 in the computer!

Set the computer IP address to either:

- 169.254.0.1, or
- 169.254.0.3.

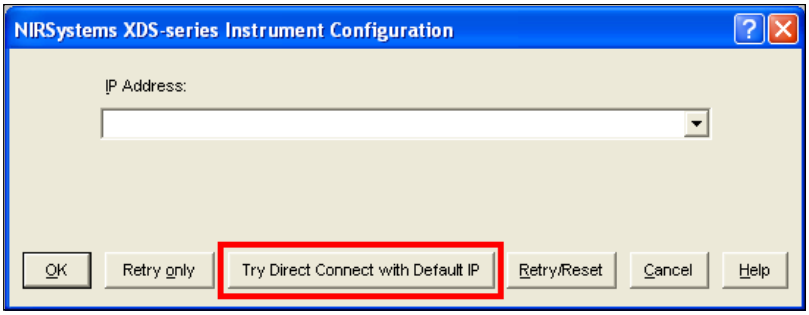


Direct Connection Solution 2: Power down the XDS Instrument, then power it back up. Wait 120 seconds, for the instrument to determine the correct method of Ethernet communication. Please do not click anything for this amount of time, or communication may be interrupted.

Direct Connection Solution 3:

Click on "Try Direct Connect with Default IP". This searches for the default IP address stored in the instrument.

When the instrument is found, click "OK"



This should resolve the connection issue. Proceed to "Acquire" and "Connect" in Vision.

4 Assembly of the Instrument

The XDS Analyzer will be assembled and installed by a trained representative of Metrohm. This person will perform a full suite of diagnostics to verify correct operation, and will explain basic operating points. Assembly information is given as a guide for the user, should re-assembly ever be required due to an instrument move or for other reasons.

Verify that the following items have been received in good condition:

- Metrohm XDS Monochromator
- Smart Probe Module (fiber length is either 2 meter or 3 meter)
- Vision Spectral Analysis Software
- Spare Lamp (in Accessory Kit)
- Accessory Kit, containing cords, cables and other required items
- Safety Manual for CE certification
- Instrument Test Results Packet

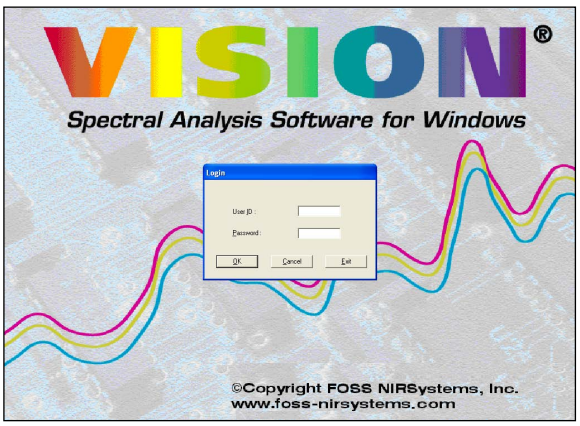
* Packaging may vary from that shown above

The serial number of the instrument and module are located on serial plates on the left side, when facing the instrument. These serial numbers should match the serial numbers marked on the shipping papers.

When using Vision software, the software will automatically read the monochromator serial number. The module serial number must be entered manually, and is located on the module serial number plate on the left side of the module, facing the instrument.

Follow the assembly sequence that begins on the next page:

1. Load Vision Spectral Analysis Software onto the computer designated to operate the XDS instrument.



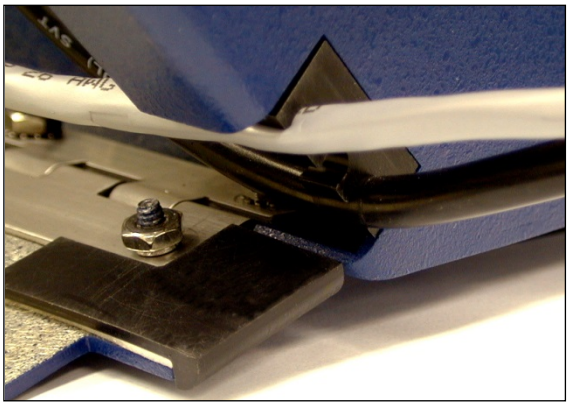
2. Place the monochromator on the lab bench in the position shown.

3. Open the right-hand panel of the instrument. Pull it gently by a fin, until the catch releases. This panel opens to about a 45-degree angle for access to connectors, and for filter inspection. Avoid scratches or damage.

4. Gently thread the AC power cable and network cable through the lower right corner of the instrument access area as shown.

   The cables should snap into the black holder. The innermost position is large, to fit the power cord. If the power cord is in the wrong location, the door may not close fully.

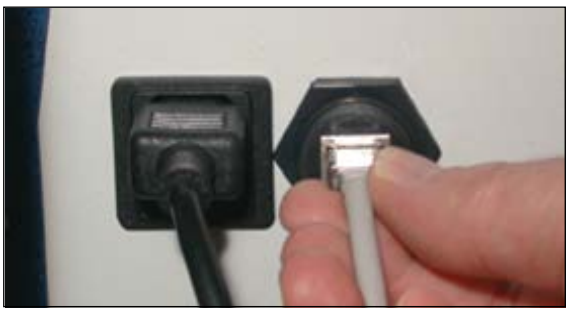
   The network cable may go into either of the other two locations.



5. Insert the AC power cable into the AC power block as shown.

6. Attach the RJ-45 cable to the network connector on the instrument. If using Direct Connection, use the gray cable from the instrument accessory kit.

   If using network connection, do not use the gray cable, as it is a "UTP crossover" cable and will not work with a network. Use a network cable as described in section 3.0.

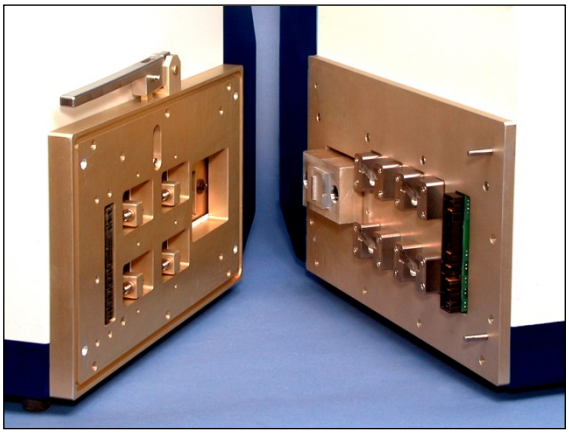


7. Close the outer cover of the instrument. Push gently to the final closed position. It should latch securely.

8. Position the sampling module directly in front of the monochromator.

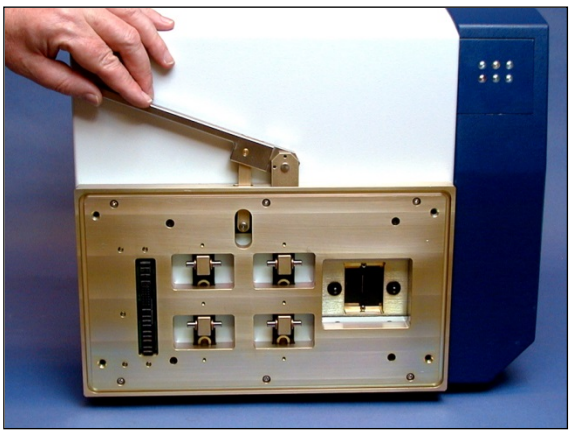
   This photo shows the mating connection plates, prior to final alignment and assembly. The locating pins help find the final position.

   The latches are used to lock the module in position.



9. Lift the release handle on the monochromator and engage the module "catches" to the locking togs on the monochromator. (Module not shown to allow a good view of handle.)

   Push monochromator and module together firmly (with handle up) then lower the release handle.



10. When the catches are fully engaged to the locking togs, push the release handle down all the way.

    This automatically engages the electrical connector and fiber optic interface, and maintains proper alignment of the module to the instrument.

    Carefully wind the fiber bundle clockwise around the "saddle." The 2-meter probe takes one wrap. The 3-meter probe (shown) takes 2 wraps.

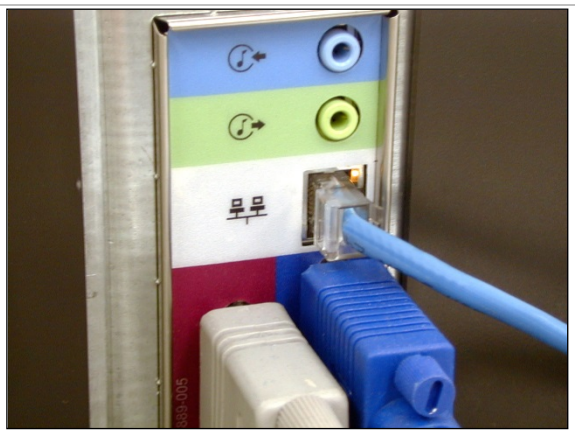
    The final assembly is as shown.

11. Plug the AC power cord into a grounded AC outlet. A surge protector or Uninterruptible Power Supply (UPS) is recommended for best operation.

12. For "Direct Connection" the gray cable from the instrument plugs directly to the computer network jack. (Use the cable supplied with the instrument.)

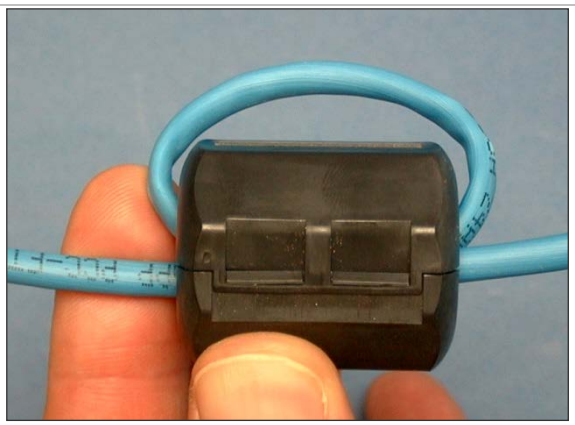
    If using a network, use a non-crossover type cable as listed in section 3.0. Plug the RJ-45 network connector into a functional network port.

    If this requires approval from a network administrator, it should be properly approved for hookup.



13. Install the ferrite onto the RJ-45 cable as shown. This is detailed in the sheet attached to the ferrite.

    This ferrite is required for CE Emissions (EMI) compliance. All users in the European Community are required to install this ferrite.



14. When all the above assembly is finished, turn on the power switch on the monochromator. It is located on the lower surface, on the right-hand side as shown.

    The monochromator performs some initialization tests, which take a moment. Some noises will be heard as items find their initial positions. This is normal.

15. Prepare to establish communication from Vision to the XDS instrument. This is detailed in Section 6.0, Vision Software.

This completes assembly of the Smart Probe Analyzer.

5 Smart Probe Module

The XDS™ Smart Probe Analyzer is designed for sampling of many types of customer samples. This section describes removal of the protective barrel, functions of the LED indicators, use of the trigger, and handling of the fiber bundle.

The Smart Probe is shipped with a blank end, to protect the fiber optic termination.

Unscrew this end and remove it.

DO NOT ATTEMPT TO OPERATE THE INSTRUMENT WITH THIS PROBE END.

Place the end into the wooden box, which contains the Interactance Reflectance and Interactance Immersion ends.

If the Smart Probe is ever shipped to another location, the blank end should be used to prevent damage.

The proper sampling end should be installed, as described in these sections of the manual:

Interactance Reflectance: 5.1

Interactance Immersion 5.2

Always be sure the barrel is screwed down fully before sampling. The black O-Ring should not be visible when fully assembled.

Failure to tighten the probe barrel will cause the instrument to fail diagnostic tests, and will cause spectral distortion.

LED Indicators on the Smart Probe Handle

Pass:
The sampled material has passed the ID and Qualification test (Routine Analysis)

Fail:
The sampled material has failed the ID or qualification Test (Routine Analysis) This means the material was either not identified, or was outside of acceptance limits for that material.

Ready:
The Smart Probe is ready to take a sample scan.

Scan:
Scanning reference or sample.

Enter:
Not used

Ref:
System is ready to scan reference material

Upon initial instrument power-up, the LED indicators will illuminate in a clockwise pattern, for 5 times. This occurs about 25 seconds after power-up. Following this, the "Module" LED illuminates. This indicates that the instrument has verified the Smart Probe as being present and operating.

When using the Smart Probe, Vision will prompt the user to pull the trigger to start the scan.

This shows the trigger. A short, decisive squeeze is all that is needed. As soon as Vision recognizes the action, the dialog box is removed from the screen.

The operator will normally wear protective gloves to meet cGMP regulations. The glove was omitted in this photo for clarity.

The fiber optic bundle, while well-protected, should always be handled with care. The individual fibers are quite thin, and can be broken by rough handling. Breakage of these strands will reduce the light energy, and will eventually cause the instrument to fail diagnostic tests.

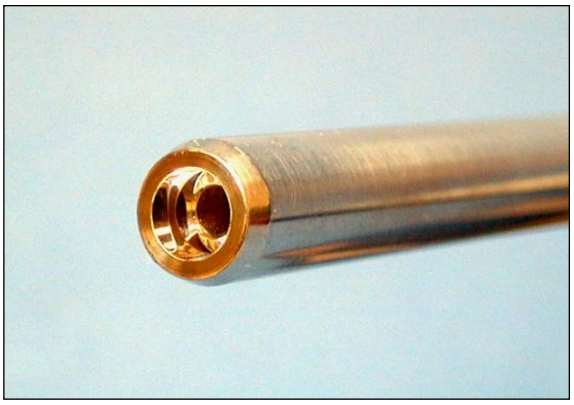
Always observe these precautions:

- Never bend the bundle tighter than a 12" (305mm) diameter. As a rule, do not bend the fiber any tighter than the gentle bend that it assumes in a normal repose.
- Always store the bundle on the holder when not in use.
- Never attempt to "stretch" the fiber beyond its reach.

Fiber bundle lengths are 2-meter and 3-meter.

5.1 Interactance Reflectance Configuration

The Interactance Reflectance barrel should be screwed onto the probe for reflectance sampling.

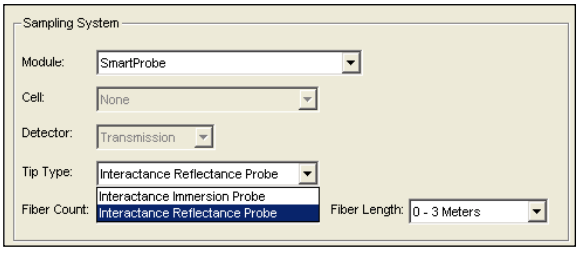


This photo shows the sample window, which is faceted to illuminate the sample, and to channel reflected energy back to the detectors.

Always be sure the probe barrel is screwed tightly, against the metal stop. Do not scratch or damage the Smart Probe window.

Select the proper Tip type in the Data Collection Method (DCM) in Vision.

(See section 6.0 for full information.)



The reference scan is taken with the Interactance Reflectance Tip located in the uppermost aperture on the front of the instrument, as shown.

Click on the reference icon in Vision. Position the probe fully into the aperture and squeeze the trigger when instructed.

With the Interactance Reflectance probe barrel mounted, the operator may sample powders in shipping containers, barrels and drums.

The 2-meter fiber length is generally sufficient to permit sampling right on a pallet or cart.

The probe barrel can be rinsed in a cleaning solution, then rinsed with distilled (or de-ionized) water to prevent sample carry-over from one barrel to the next.

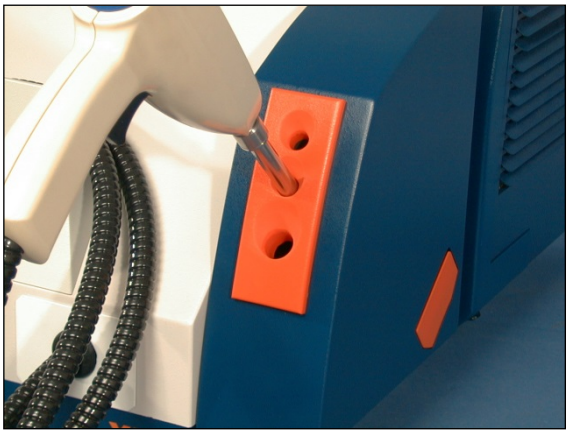
This should be written into the cleaning procedure for the instrument. In the pharmaceutical industry, a "cleaning validation" may be required.

NOTE: Any dirt or material carried on the probe barrel may soil or damage the instrument reference. This should be avoided!

When finished sampling, the probe should be stored in the reference position. This is the proper position for Performance Test, Gain Test, and Wavelength Linearization.

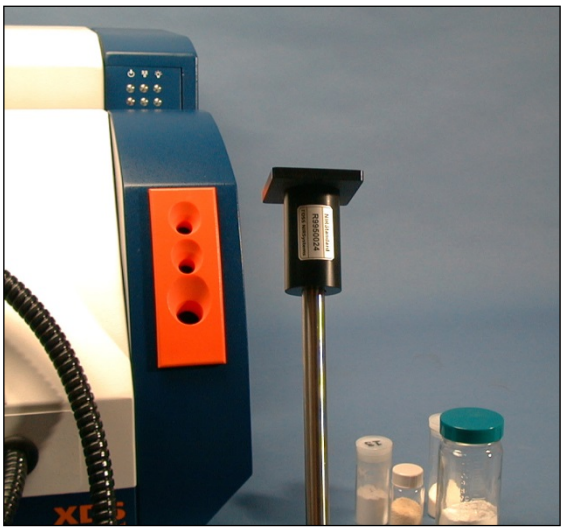
Instrument Calibration makes use of an external wavelength reference. The reference mounts in a cup holder on the side of the instrument as shown. (Leave room for easy access.)

Please see the section on Instrument Calibration for full information.



Reference Standardization requires use of an XC-1200 Calibrated 99% Reflectance Reference.

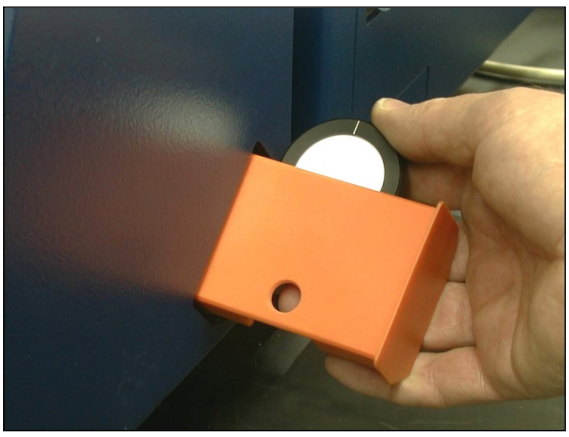
Do not attempt to perform this with the R99 from the XC-1010 Standard Set. Please see the section on Reference Standardization for full information.



When instructed to scan the Wavelength Standard, or the Calibrated Reference Standard, simply press the door, and the holder will open.

Pull as far as it will come out, then insert the cup, window side toward the probe.

Close door and insert the probe into the middle aperture, against the sample cup.

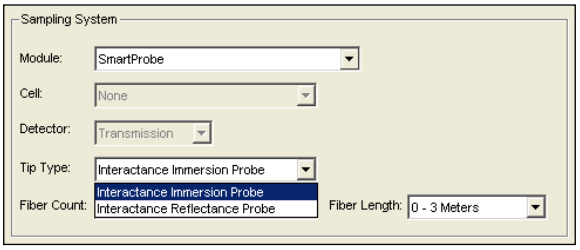


5.2 Interactance Immersion Configuration

This is the Interactance Immersion (Liquid) Tip used to sampling liquids such as alcohols and other clear to semi-clear liquids. The path length of the tip is set by use of a spacer, as shown in a later frame.

Select the proper Tip type in the Data Collection Method (DCM) in Vision.

(See section 6.0 for full information.)



Spacers are available for 2, 4, 5 and 10mm.

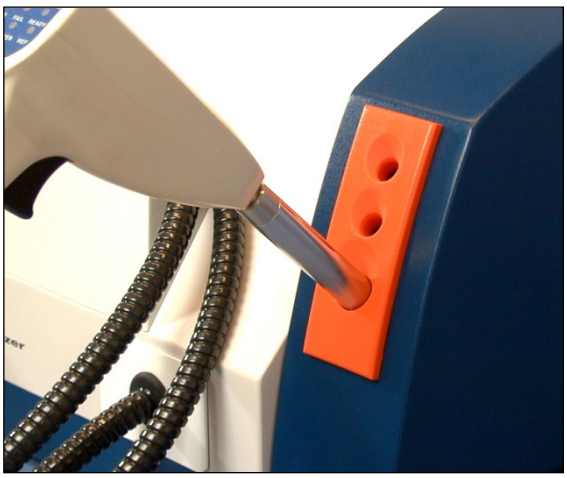
Note that the pathlength through the liquid sample is 2X that of the spacer, since the energy is transmitted through the sample to the mirror, then back through the sample to reach the window to carry the signal to the detectors.

The spacers give pathlengths as shown:

- 2mm spacer 4mm pathlength
- 4mm spacer 8mm pathlength
- 5mm spacer 10mm pathlength
- 10mm spacer 20mm pathlength

The reference scan is taken with the Interactance Immersion Tip located in the lowermost aperture on the front of the instrument, as shown.

Click on the reference icon in Vision. Position the probe fully into the aperture and squeeze the trigger when instructed.



When placing the Interactance Immersion (Liquid) Tip into the sample, angle it slightly to avoid trapped bubbles between the mirror end and the sample window. Bubbles will change the spectral readings.

Cleaning is similar to the Interactance Reflectance end. Use a suitable cleaning solution, then rinse with distilled water to prevent sample carry-over from one sample to the next.

When finished sampling, the probe should be stored in the reference position. The probe end is protected from ambient light in this position. The end should be clean and dry when placed into the holder, to avoid drips and puddling inside the assembly. A small drain hole is provided, primarily to protect the instrument from accumulated drips.

This is the proper position for Performance Test and Gain Test using the Interactance Immersion (Liquid) End.

Reference Standardization is not technically required for the Interactance Immersion tip, because air is used as a reference, and it is assumed to be 100% transmissive.

However, Reference Standardization is required for Instrument Calibration, and should be selected in Configure, Options.

Instrument Calibration must be performed in the Reflectance mode. After mounting the Interactance Immersion tip, run Wavelength Linearization if Auto-Linearization is selected under Project Options.

Always follow these guidelines when handling sample materials:

1. Always use appropriate safety equipment with hazardous materials, to prevent injury. This equipment may include safety glasses, gloves, lab apron, and fume hood. Always check the MSDS (Material Safety Data Sheet) for the materials being analyzed for recommended handling precautions.
2. When inserting the reflectance tip into powdered samples, try to press it consistently each time. This provides fairly uniform "sample packing" in front of the sample window, and gives minimal baseline differences in spectra.
3. Be sure the probe end is not at the extreme edge of the container, where sample material may be thin. The probe should always see "infinite thickness" of powder for best operation. This varies with material, but 13mm or 1/2" is generally considered sufficient.

Cleaning the Probe End:

Following analysis, the probe end should be cleaned to prevent sample carryover, which could affect analysis. Cleaning depends upon the sample material. Always follow the precautions on the MSDS when performing cleaning. Normal samples and cleaning methods include:

Sample Type	**Cleaning Method**
Free-flowing crystalline materials, pellets, fibrous samples	Wipe from probe end with disposable lab wipe. Wipe probe window with dampened wipe. Rinse with de-ionized water, and dry thoroughly.
Dense powders which tend to clump, thick slurries	Wipe from probe sampling area with spatula or wooden tongue depressor. Clean window using a mild detergent solution, rinse, then dry.
Thick, pasty samples with high oil content	Dig out using spatula or wooden tongue depressor. Wash cell and window in a strong detergent to remove all residual sample material. Rinse and dry.

Pharmaceutical applications may require a "Cleaning validation" which describes each step of the cleaning process, solvents to be used, and specific verification tests to determine whether cleanliness

levels have been reached. Because a cleaning validation is sample-specific, Metrohm cannot recommend detailed cleaning methods for such samples.

5.3 Cleaning of internal reflectance reference

The internal reflectance reference is a small piece of 99% reflectance (nominal) Spectralon™. This material is quite rugged, non-hygroscopic, and stable with temperature.

In spite of these qualities, the internal reflectance reference can become contaminated. This chiefly occurs through foreign materials becoming lodged on the end of the Smart Probe, then deposited on the internal reflectance reference when the probe is placed into the reference position.

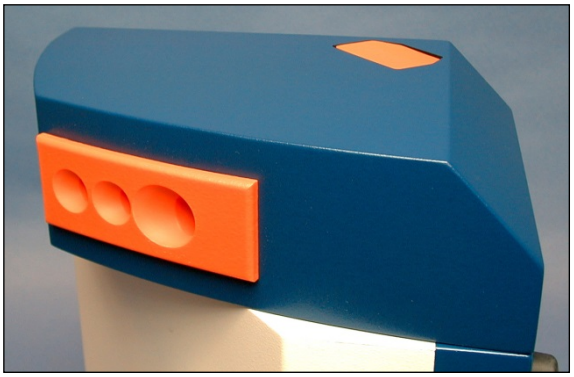
It is important to always clean the Smart Probe tip before placing into the reference position. Never leave any sample material on the end. It is wise to clean the end thoroughly, then wipe dry with a non-linting laboratory tissue or other type of wipe.

To clean the internal reflectance reference, follow these steps:

1. Remove the Smart Probe from the reference position, and place it in a secure place where it will not fall or be damaged.

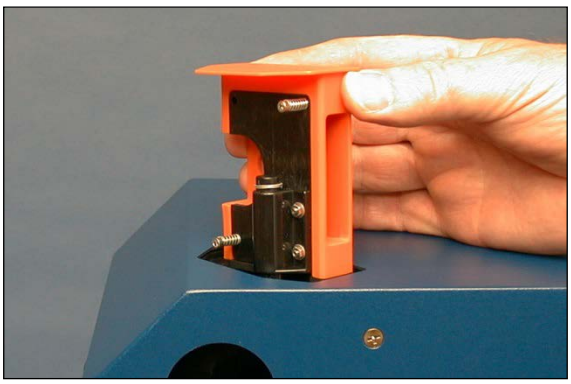
2. Lift the release handle, then remove the Smart Probe module from the monochromator.

3. Gently lift the module onto its left side, with the blue housing upward.



4. Gently pry the large black cap off the back of the module as shown.

5. Lift the sample drawer upward, out of the way. (Hold in this position.)



6. Using a large flat-bladed screwdriver, loosen the large (3/4") threaded plug inside the instrument opening.

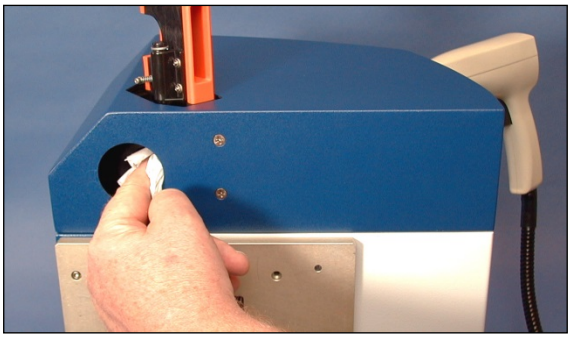
   A larger screwdriver than the one shown is preferable. A small screwdriver is shown to give the best view of the threaded plug.

   When the plug is loose, hold the screwdriver blade horizontal (as shown) to lift the threaded plug out of the unit.

7. Remove the plug.

8. The white object inside the opening is the reflectance standard.

9. Reach in with a tissue, then press the reflectance standard out from the other side -- insert the Smart Probe gently into the reference position. The standard should pop out into the tissue.

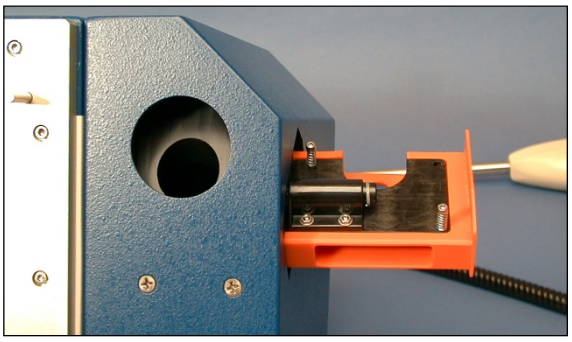


10. Gently wipe the surface with a laboratory tissue to clean it. Do not abrade or pit the surface in any way.

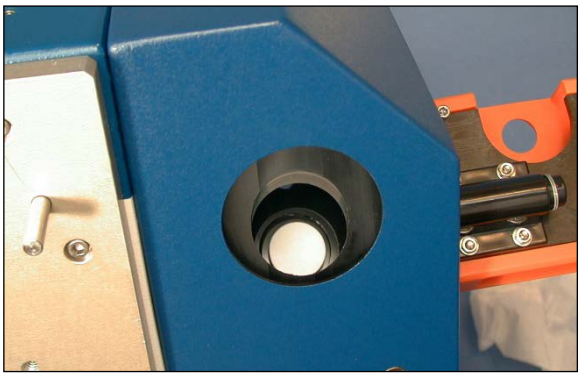
11. To re-install the reflectance standard and the threaded plug, gently turn the Smart Probe over onto the face of the module.

    Place a towel or pad underneath to avoid damaging the paint. Be very careful of the fiber optic cable when placing in this orientation.

    In this position, the standard can be inserted with minimal handling.



12. When the reflectance standard is clean, re-insert it into the instrument opening. Handle by the edges to avoid deposition of oils onto the surface.



13. Re-install the threaded plug and snug down.

14. Slide the sample drawer back to the normal position.
15. Install the Smart Probe module back onto the monochromator, and tighten the release handle.
16. Place the Smart Probe into the reference position.
17. Warm up the instrument thoroughly. Run Performance Test to verify proper operation.

    (See section 7.2 for test instructions and information.)

18. Run Reference Standardization to create a new calibration for the newly-cleaned internal reflectance reference.

    (See section 7.1 for test instructions and information.)

Always protect the Certified 99% Reflectance Reference from contamination:

1. Always clean the tip of the Interactance Reflectance probe thoroughly before performing Reference Standardization.
2. Store the Reference Standard in its protective case between uses.
3. Avoid any introduction of dirt or contaminants into the surface of the Reference Standard. Dirt and sample carryover will change the optical characteristics of the standard, which will then affect how the instrument reads samples.

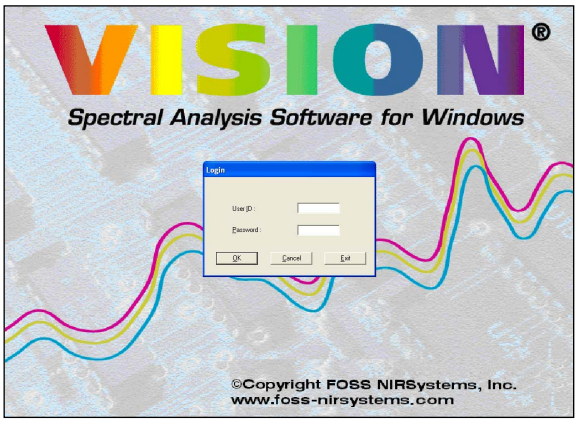
The quality of the instrument spectra depend upon proper Reference Standardization. This XC-1200 Reference Standard has been calibrated on a Master Smart Probe instrument which reads on a scale of true reflectance. The calibration for the Reference Standard is the linkage back to traceability, and is essential for good method transfer with the Smart Probe.

6 Vision Software: Connection to the Instrument

This section describes communication between the computer (with Vision Software loaded) and the XDS instrument. Please follow these steps to establish communication. The instrument may be connected as explained in section 3.0 of this manual. The preferred method is that the instrument and computer both be plugged into live RJ-45 communication jacks, on an active network.

Install Vision on the computer to be used for instrument operations.

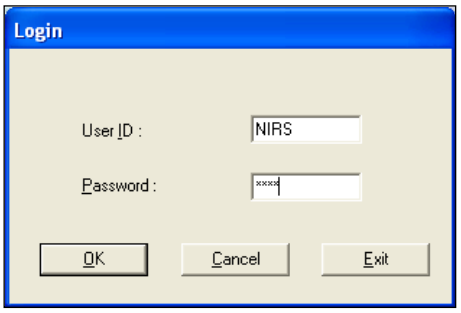
Once installed, click on the Vision icon on the desktop. The log-in box appears on the opening screen.



Enter the default User ID, "NIRS". It is not case-sensitive.

Tab (or mouse) to the Password box, and enter the default password, "NIRS".

Note that you should set up specific User ID and passwords for each authorized user. Do not operate on the default User ID, or you may be in violation of CFR 21, Part 11.

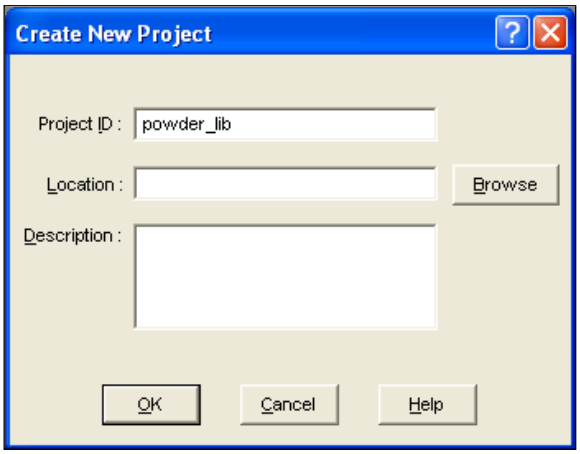


To begin, a new project must be created. The project is used to store data and calibrations for a given type of analysis.

Multiple projects may be used, to keep spectra, calibrations and other data separate and well-organized.

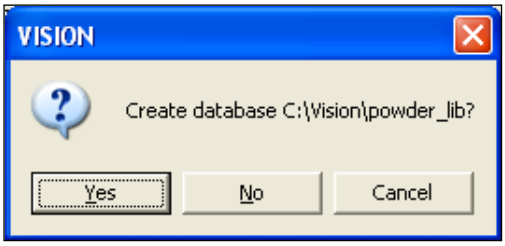
Assign the project some meaningful name, to make it easy to remember. Do not use spaces – use an underscore instead, as shown. The database requires that there be no spaces used.

Vision will assign a Location; leave this blank.



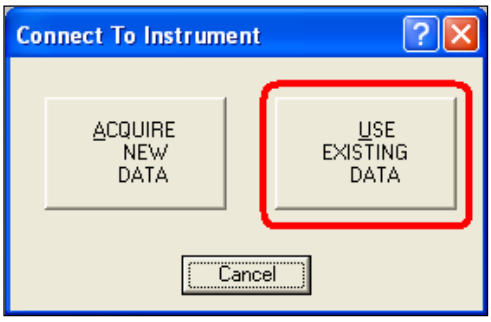
Vision asks if the default directory location is acceptable.

Click on “Yes”. Vision creates a directory for the project as shown.

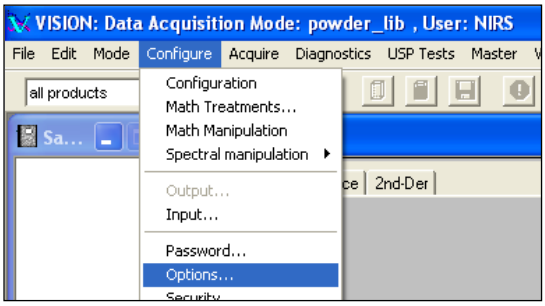


Vision prompts the user to connect to the instrument. Be sure the instrument is turned on and is ready.

Click on “Use Existing Data” to postpone instrument connection. The Project Options must be verified before connecting.

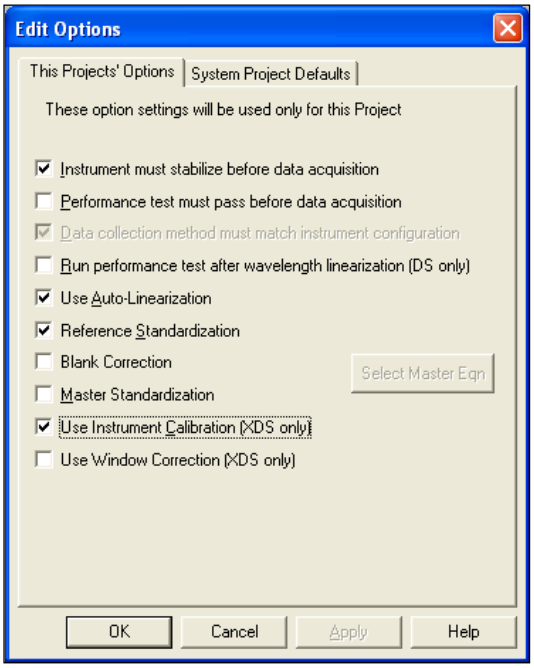


Click on “Configure”,” Options” to set Project Options.

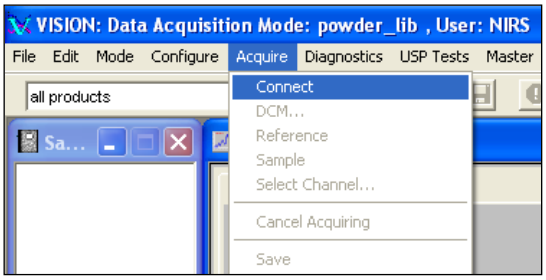


Project Options should be set as shown. If they are not this way, click on the “checkbox” to select or de-select as needed.

When the Project Options are as shown, click on “OK”.

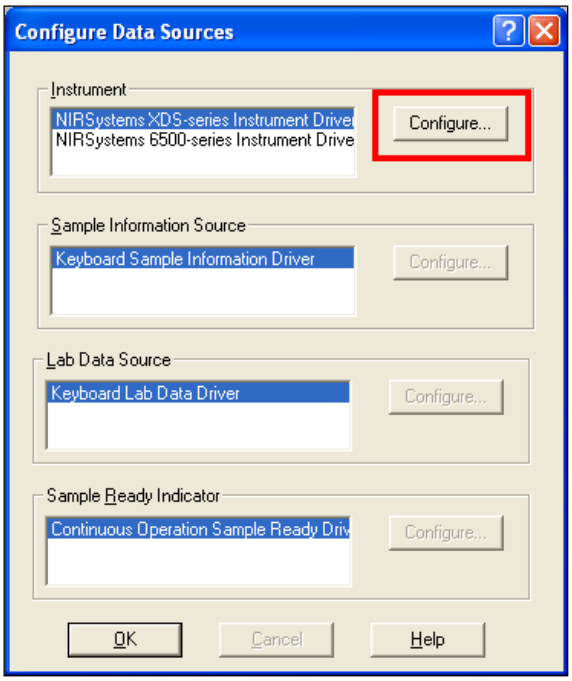


Click on “Acquire”,” Connect” to initiate instrument connection.



This screen sets up communication parameters for the instrument. The XDS instrument has a unique driver.

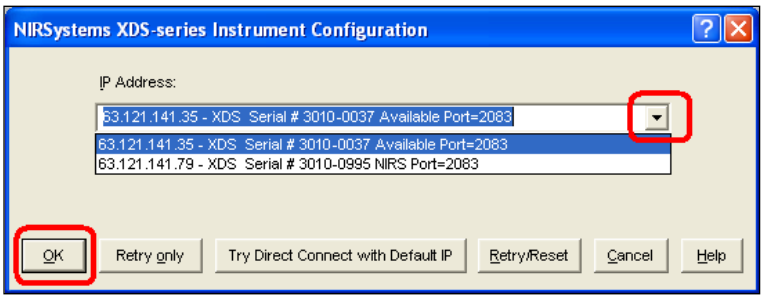
Highlight “NIRSystems XDS-series Instrument Driver” and click on “Configure”.



This box allows the user to select the instrument by serial number. The IP (Internet Protocol) address is also shown.

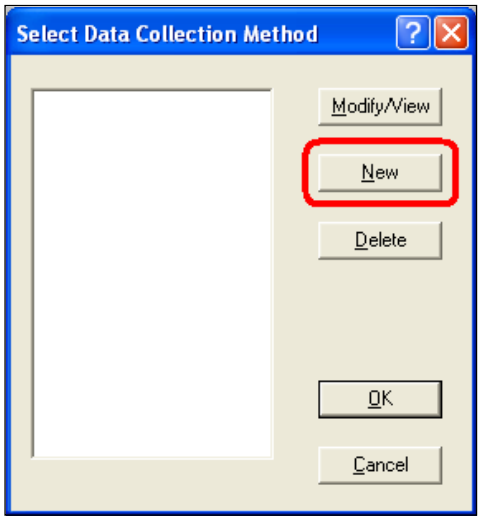
Use the drop-down arrow of the IP Address box, and select the correct instrument. The instrument must show “Available Port=2083” to be selected.

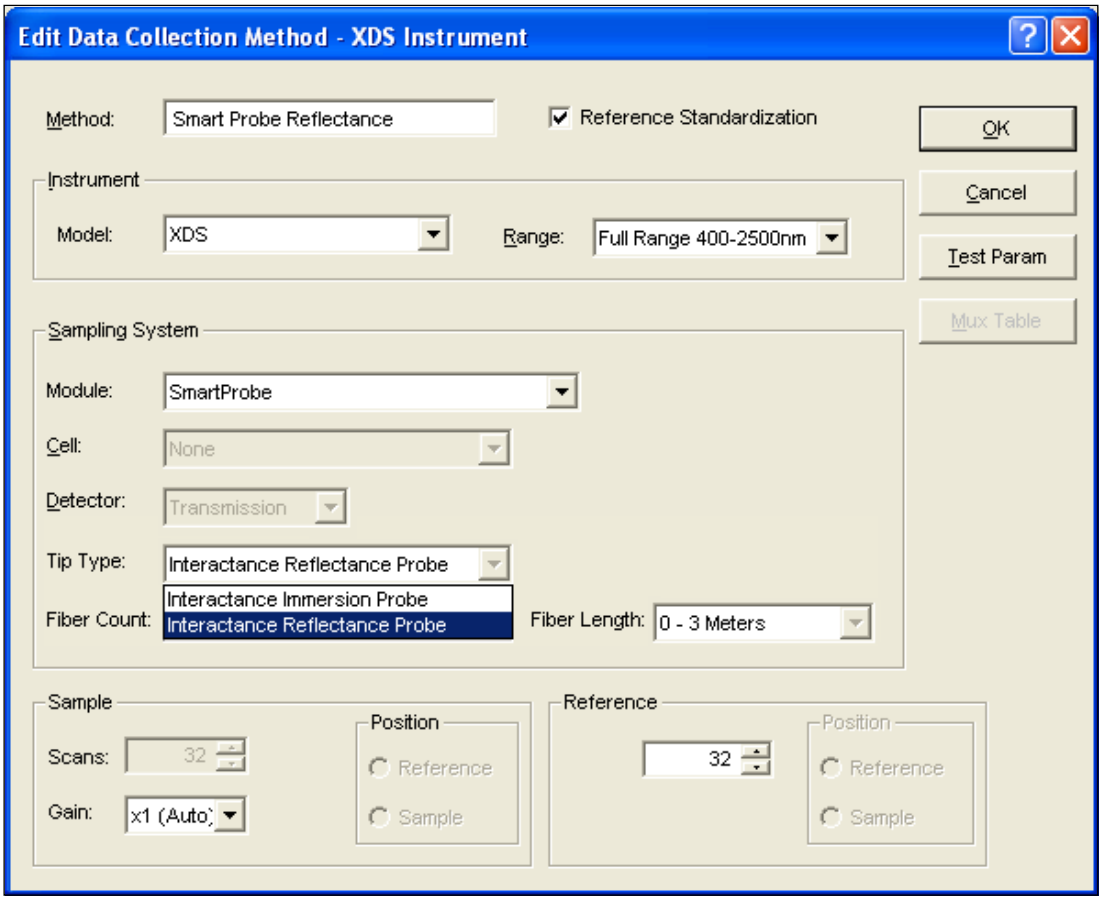
Click “OK” to exit the dialog box.



A Data Collection Method (DCM) must be established to communicate with the instrument.

There are no Data Collection Methods available upon initial connection. Click on "New".





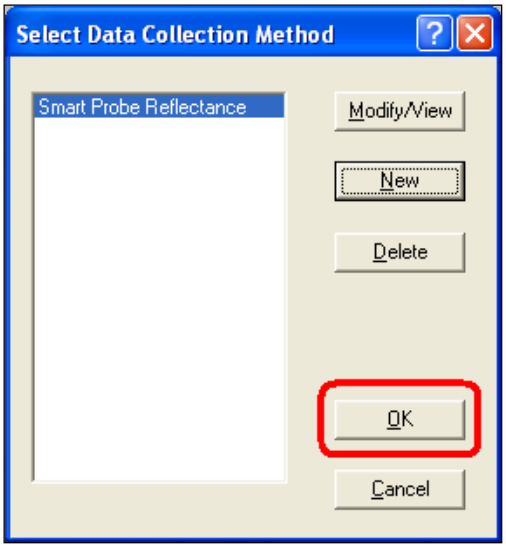
The instrument "self-identifies" as an XDS system. The DCM will self-select as a Smart Probe Module. Select the proper probe type, either Interactance Reflectance, or Interactance Immersion. The user should enter a logical name for the DCM, such as that shown above.

Use the default of 32 scans for "Sample" and 32 scans for "Reference." The instrument scans on each forward swing and each backward swing of the grating, unlike previous Metrohm instruments. Thus, 32 scans are accomplished on only 16 grating cycles, and are very rapid. Click "OK" when finished.

The user will hear a slight ticking sound from the internal order sorter whenever the lamp is on. This is normal, and has no effect on component life. The parts are kept in motion to reduce "wait time" before instrument stabilization.

Next, select this DCM from the selection box. Click “OK” to connect to the instrument.

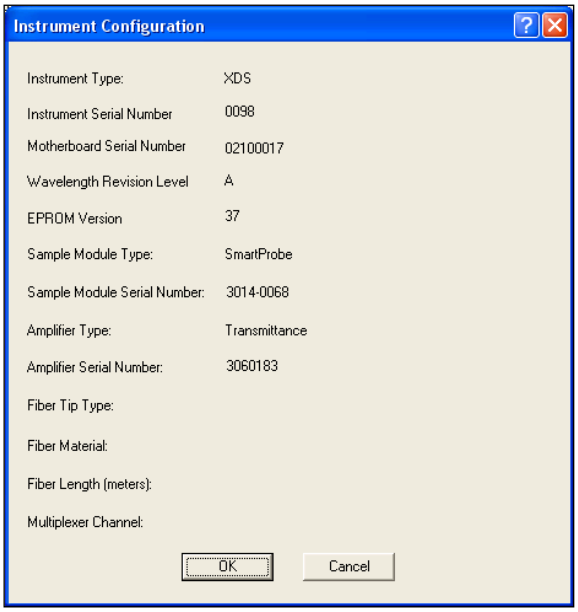
Once connected, verify that the amber “Communication” LED is lit on the instrument



Upon connection, Vision will prompt for the instrument configuration. This is used to establish a Diagnostic Database.

It is important that the instrument be correctly identified, to prevent corruption of the database.

If only one instrument is in use, accept the information and click “OK”.

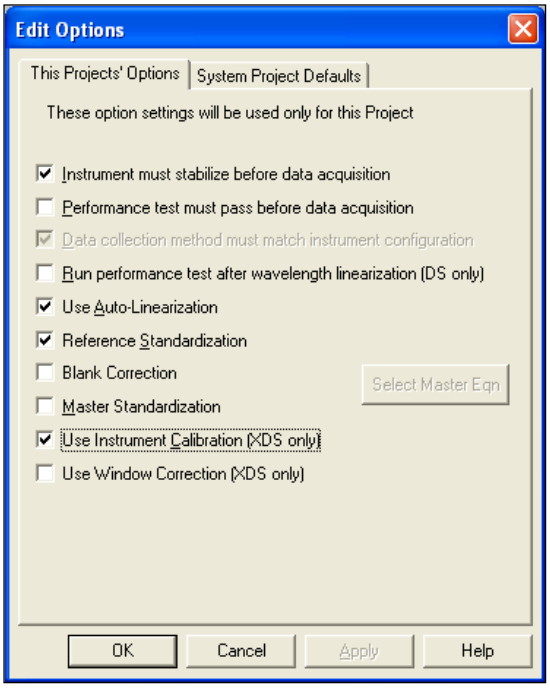


7 Instrument Diagnostics

Vision provides diagnostics for instrument setup, which must be performed before use of the instrument for analysis. Following these diagnostics, another set of diagnostics is provided to evaluate the ongoing performance of the instrument. These are explained in the sections that follow.

7.1 Setup Diagnostics

Before use of the XDS Analyzer, some steps must be performed using Vision Software.



The "Options" for a given Project may be set. To reach this menu, click on Configure, Options. The menu shown at right is the default set of selections. A brief explanation follows:

Instrument must stabilize before data acquisition: This prevents spectral acquisition if the instrument is cold.

Performance Test must pass before data acquisition: This prevents the user from taking data on a non-functional instrument.

Data Collection Method must match instrument configuration:
Checked by default to prevent incorrect instrument selection.

Run performance test after wavelength linearization: Forces user to run test sequentially. This is not necessary with XDS.

Use Auto-Linearization:
Maintains correct wavelength registration automatically, using internal wavelength materials to keep instrument in precise adjustment over time.

Reference Standardization:
Used to create a virtual 100% reflectance reference, using a traceable photometric standard. This is explained in the next section. Required for Method Transfer.

Blank Correction:
Not used on this model – applies only to XDS Rapid Liquid Analyzer.

Master Standardization:
This method is not used with XDS. Do not select.

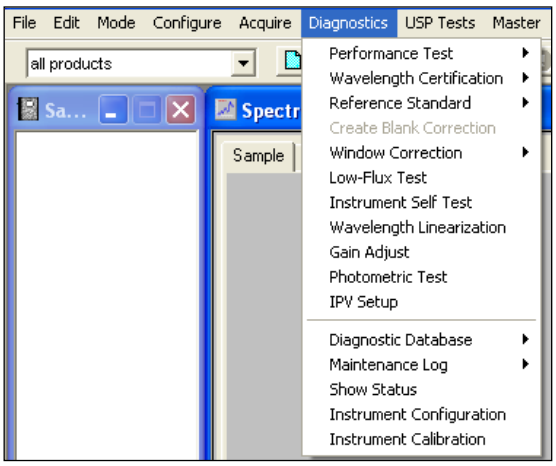
Use Instrument Calibration (XDS only)
This is a method to adjust the instrument wavelength profile to an external, traceable wavelength standard. It is checked as a default for XDS. Required for Method Transfer.

Use Window Correction:
This is used only on certain XDS Process Instruments. Do not select for XDS Smart Probe.

When method transfer (between instruments) is anticipated, be sure that Reference Standardization and Instrument Calibration are selected. These features assure best method transfer between similar instruments.

Some of these selections are spectroscopic calibrations, and are used to apply corrections to the instrument to minimize differences between units of the same configuration.

The correction programs are accessed from the Diagnostics menu bar, shown at right. The diagnostic steps are explained in the sections that follow.



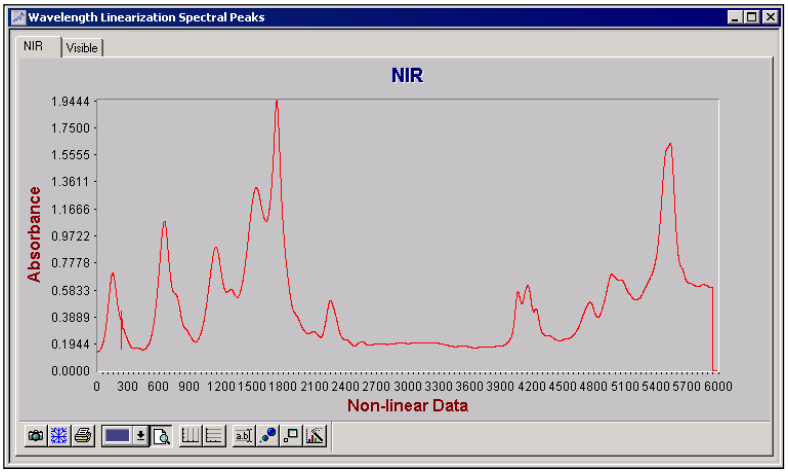
7.1.1 Wavelength Linearization

Wavelength Linearization uses an internal wavelength standard set to determine a set of internal, arbitrary peak positions that the instrument will use to maintain repeatability of wavelength response.

The NIR wavelength positions of these peaks appear as shown.

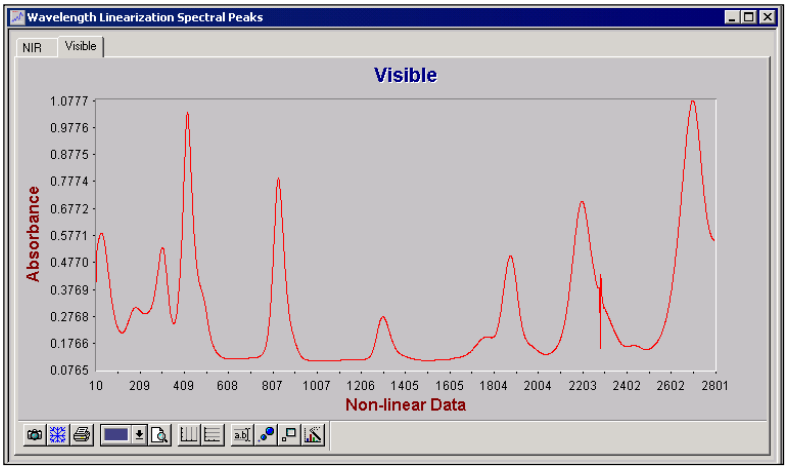
The scale of this display is marked in encoder pulses, which do not relate to nanometers directly.

From the peaks, a linearization is performed, which allows assignment of nanometer values.



The "visible" portion of the spectrum is similar. A linearization is applied to this portion of the spectrum.

Minor artifacts appear in these raw spectra due to detector crossover and other spectroscopic reasons. After linearization these artifacts are minimal or not evident, some being beyond the usable range of the instrument.

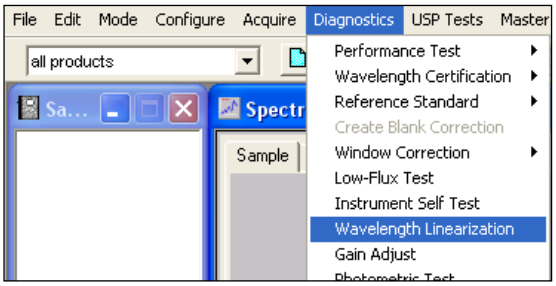


These peak positions are not meant to be traceable, as the wavelength calibration of the instrument

is done on an external standard, traceable to NIST.

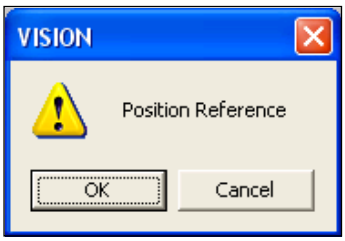
The internal wavelength materials are used to maintain the wavelength registration of the instrument. This is done by software. Final, tighter adjustments are later performed in Instrument Calibration.

Select Wavelength Linearization from the Diagnostics menu. The instrument will scan the instrument reference.



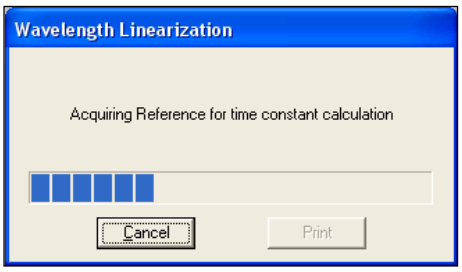
Vision prompts to position the reference. Verify that the Smart Probe is positioned as shown.

Verify the probe position and click “OK”.



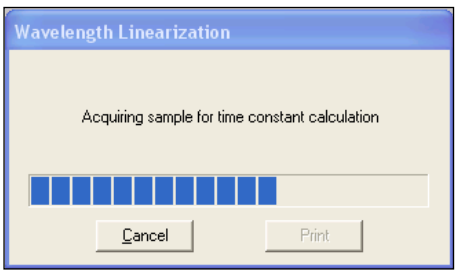
Vision acquires spectra needed to perform Wavelength Linearization.

No operator action is required at this point – this screen is for information only.



Vision continues.

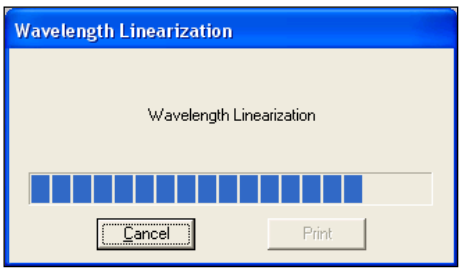
No operator action is required at this point – this screen is for information only.



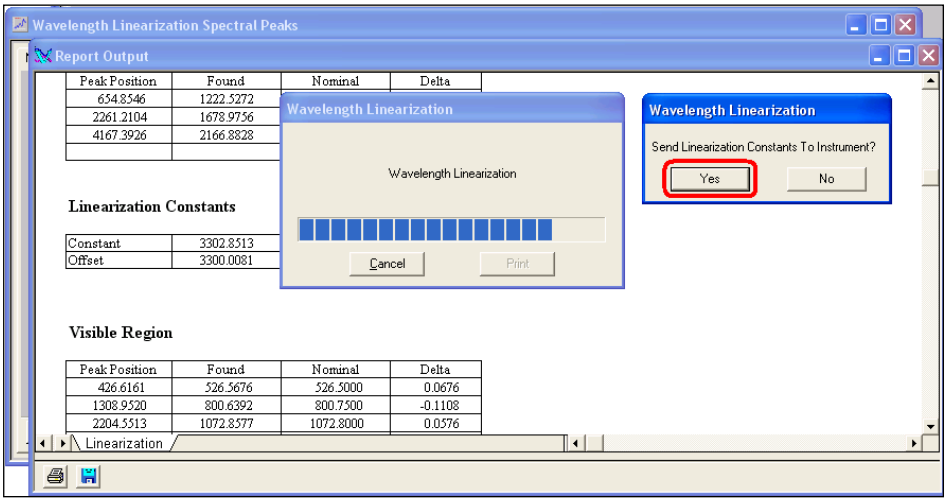
Vision continues.

No operator action is required at this point – this screen is for information only.

Once Vision has finished with this screen, a result screen will be displayed.



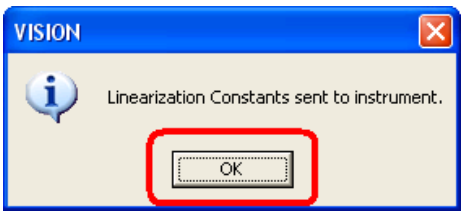
A typical result screen is shown. Peak positions for the reference materials are located using a peak-finding algorithm. These "found" peaks are compared to the nominals. Differences should be no more than 0.4nm for any peak.



Click "Yes" to send the linearization to the instrument.

After the linearization is successfully sent to the instrument, this message confirms the transfer.

Click "OK" to proceed.



This procedure is done twice, once for each direction of the grating motion. Repeat steps as prompted by Vision.

7.1.2 Reference Standardization

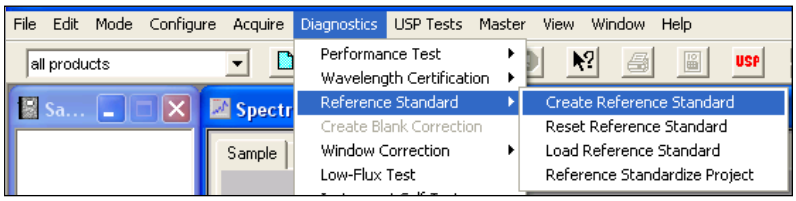
Reference Standardization is a method to provide a virtual 100% reflectance reference at each data point, to serve as a true spectroscopic reference with no character attributable to the physical reference used. This is important to achieve a high-quality spectrum on each instrument, and to enhance transferability between instruments.

A photometric standard of known reflectivity (as measured on an absolute reflectance scale) is scanned on the instrument. The instrument Spectralon® standard, located in the upper opening, is scanned. The differences of the instrument standard from 100% reflectivity are mapped, and a photometric correction is generated. This correction is then applied to every spectrum taken on the instrument, to make each spectrum appear as if taken with a reference of 100% reflectance. This assures that bright samples do not saturate the instrument, or produce negative absorbance values.

Vision software stores the Reflectance Standard file, which is downloaded to the instrument, and is

applied as a correction to each spectrum. Follow these steps to create a reference standard:

Select Diagnostics, Reference Standard, Create Reference Standard.

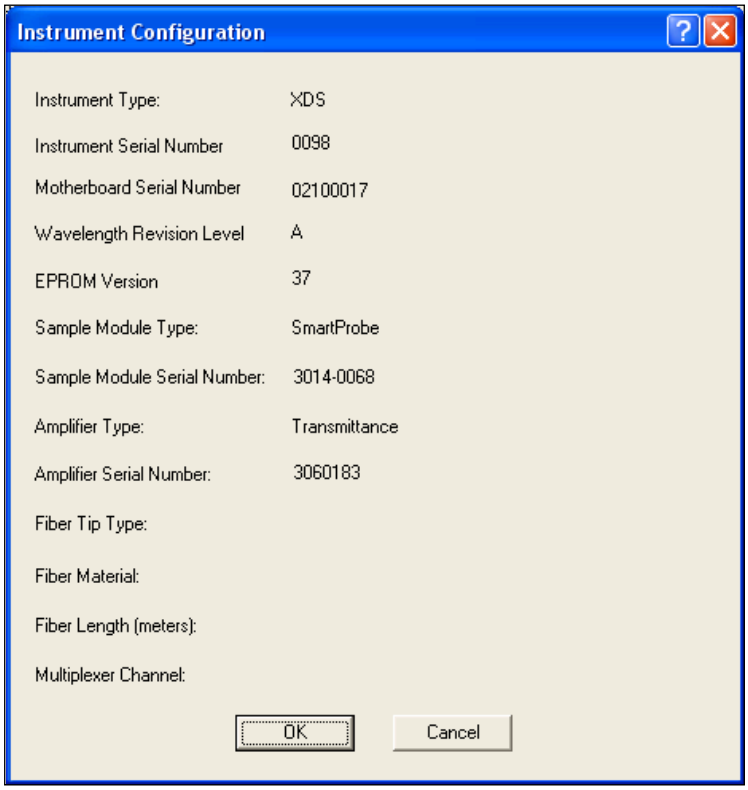


When Vision first communicates with the instrument (or after disconnection and re-connection) this screen is shown.

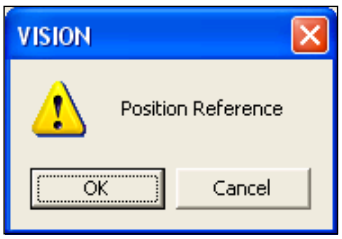
Vision must establish the configuration and have it verified by the user. This assures that test data is sent to the correct location in the Diagnostic Database.

If the Sample Module Serial Number field is empty, locate the sample module serial number on the side of the module. Record the serial number and enter it in this field.

Click "OK to accept the instrument identification.



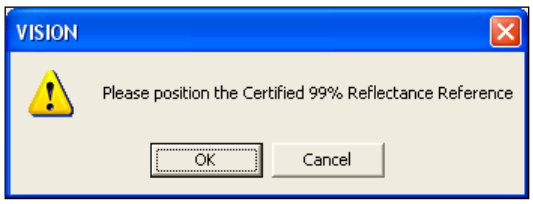
Vision requests that the user position the reference. With the Smart Probe Analyzer, the reference position is in the topmost opening, as shown in the photo below.



Place the probe in this position.

Click "OK" in the dialog box. The status bar indicates scan progress.

Next, Vision requests that the Certified Reflectance 99% Reflectance Reference be scanned.



The XC-1200 Certified 99% Reflectance Reference is shown. It comes with a mini-CD, which must be used during Reference Standardization.

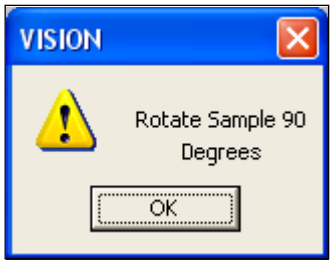
Place the standard onto the Smart Probe as shown. Do not press – simply let it rest on the probe window as shown.

Click on "OK".

NOTE: Do not attempt to perform Reference Standardization with a standard from the XC-1010 set. The XDS Smart Probe must be calibrated with the XC-1200 only.

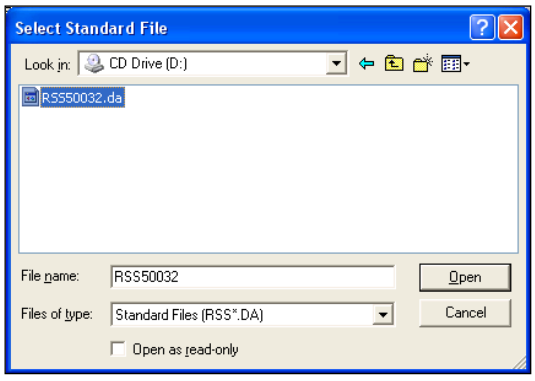
Vision prompts the user to rotate the Certified 99% Reflectance Reference. This is done to minimize the directional effects of the standard, and provide best consistency.

Note the label on the standard, and rotate 90 degrees at each prompt. A total of four positions will be used.



Vision requests the Standard File for the Certified 99% Reflectance Reference. This file is on the mini-CD packed with the standard. It may be left on the CD, or the file may be copied to the Vision directory for ease of use.

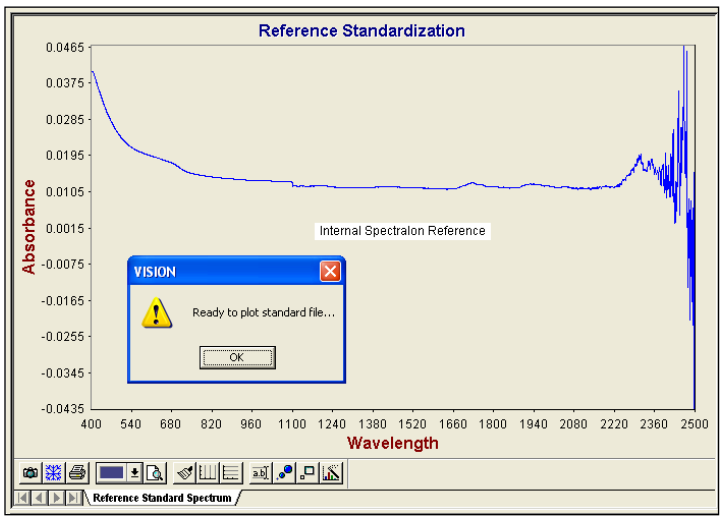
The file is named "RSS5xxxx.da". Click on the file, then click "Open".



Vision averages the spectra of the Certified 99% Reflectance Reference, as indicated by the status bar on screen.

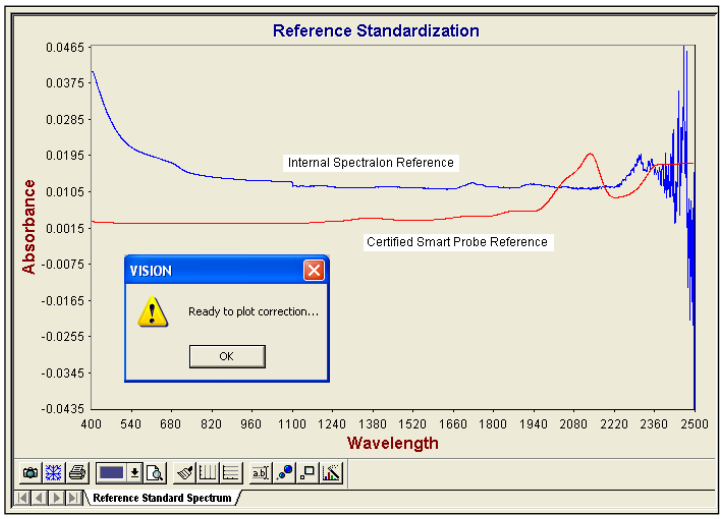
When finished, Vision plots a spectrum of the instrument reference, as shown.

Click "OK" to plot a spectrum of the Certified Reflectance Reference.

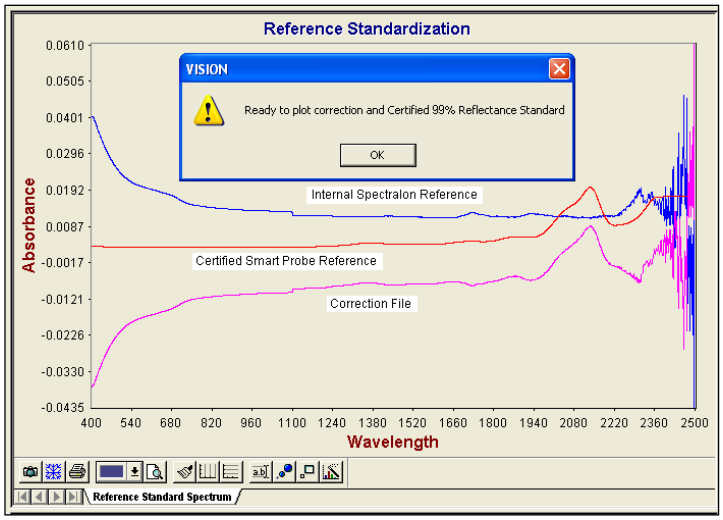


The Certified Smart Probe Reflectance Reference is shown with the spectrum of the instrument reference.

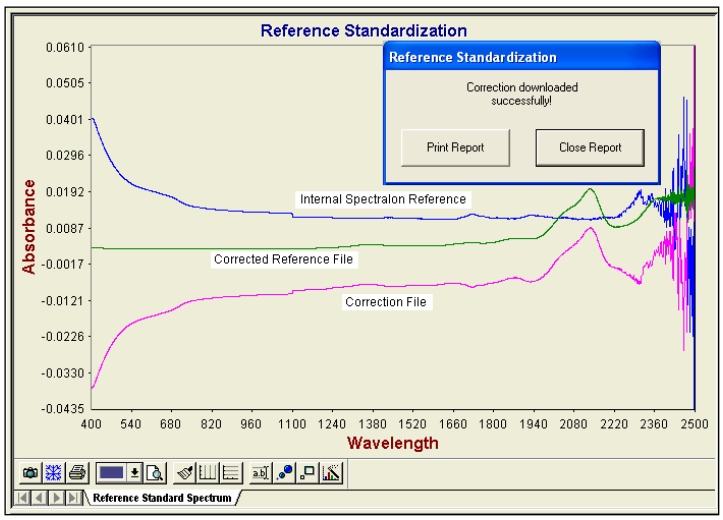
Click "OK" to plot a correction spectrum.



The correction spectrum represents the amount of spectral correction required to provide a virtual 100% reflectance reference at each data point.

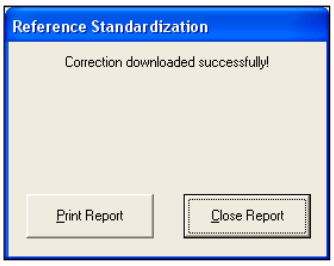


A final spectrum (green when plotted on-screen) is plotted to verify that the corrected spectrum produces the same results as the Certified Reflectance Reference.



Click "Print Report" if desired. Then click "Close Report" to continue. The correction is automatically downloaded, and is saved in the Diagnostic Data Base.

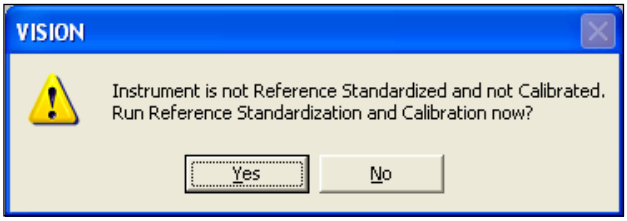
The correction will be applied in real time to all spectra taken with a DCM where "Reference Standardization" is checked.



Note that cleanliness of the probe window is very important when this program is run. If the window is not extremely clean, the character of the window contamination will be imparted to the reference correction. Therefore, maintain a clean window at all times.

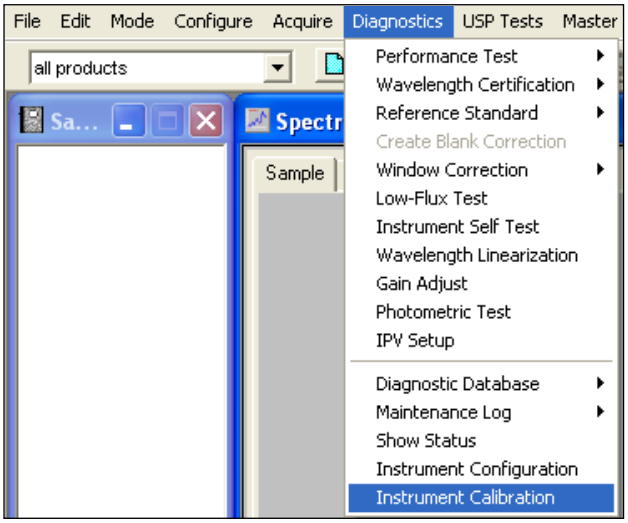
If a Reference Standardization does not exist for the selected configuration, Vision will display this message.

The user should perform both Reference Standardization and Instrument Calibration, to assure good method transfer.



7.1.3 Instrument Calibration

Instrument Calibration uses a stable rare-earth standard, of known wavelength response, as a method to establish wavelength scale response of the instrument. This standard is directly traceable to NIST SRM-1920a.



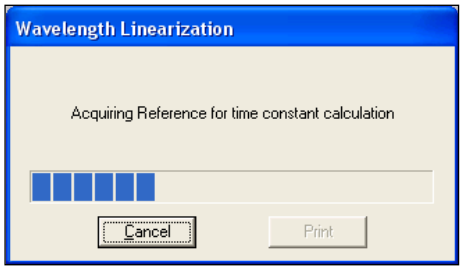
The instrument is set to scan the standard, and the nominal peak positions for each major absorption are determined. Vision performs an algorithm to set the peak positions of the instrument to those of the standard. These wavelength positions are applied at the sample plane, where samples are measured or read.

These adjustments are saved, and are applied on each subsequent scan of the instrument, yielding a correct spectrum.

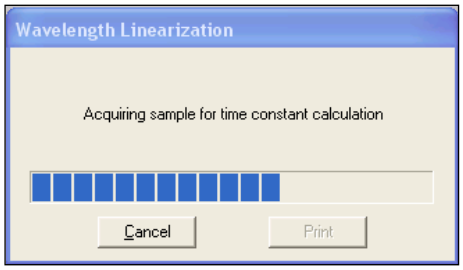
This adjustment is performed in reflectance, using the Interactance Reflectance Probe tip. Once done, this calibration applies to Interactance Reflectance and Interactance Immersion

Vision takes an instrument reference, which takes about 20 seconds. The Interactance Reflectance Probe must be in the uppermost position in the orange holder.

This dialog box is displayed.

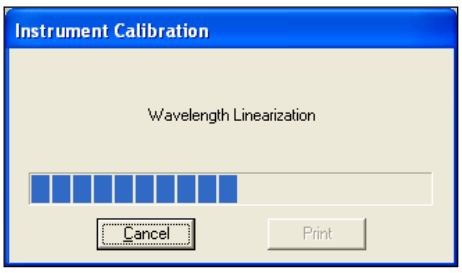


Next a sample scan is taken (on the reference) and this box is displayed. This takes about 20-30 seconds.



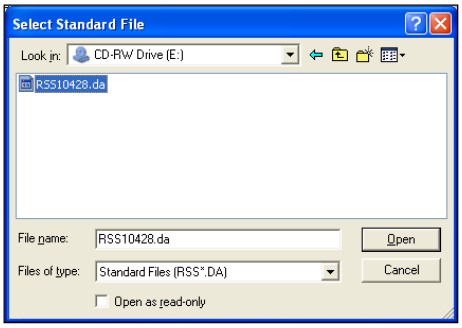
Wavelength Linearization is performed in two sections, each of which takes about 45 seconds.

The user must confirm the linearization to proceed. A dialog box will appear, and the user should click "Yes" to proceed to the next step.



Following Wavelength Linearization, the user is asked to select a standard file. This is provided on the mini-CD in the standard box. It may be manually copied on the computer in the Vision directory, as shown.

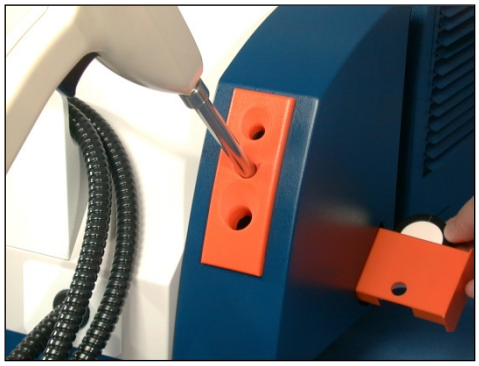
Select this file and click on "Open".



Vision prompts the user to insert the WSR10xxx standard into the instrument.

Insert the WSR10xxx Wavelength Standard Cell when prompted. Insert the cell in a consistent rotational position for repeatability.

The probe must be in the center position as shown, and the WSR standard must be in the drawer on the side of the instrument. Close the drawer first, then gently lower the Smart Probe, into light contact with the standard window. (The drawer is shown open for illustration purposes only.)

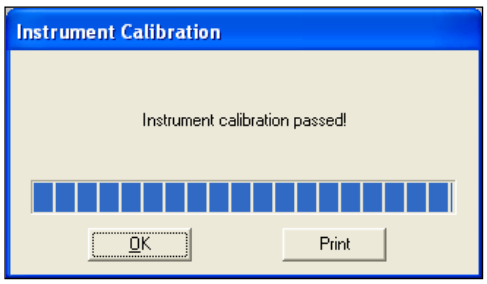


Click "OK" to continue.

This test takes about 45 seconds.

The wavelength response for each defined peak is adjusted, to assure precise wavelength registration between instruments. At the same time, bandwidth (bandpass) is measured, and is iteratively adjusted to an optimum value for the peaks measured. This is performed to assure good agreement from instrument to instrument, should multiple instruments be used for analysis of similar products.

At the end of the test this dialog box is displayed. Click "OK" to exit Instrument Calibration.



Information about the wavelength standard:

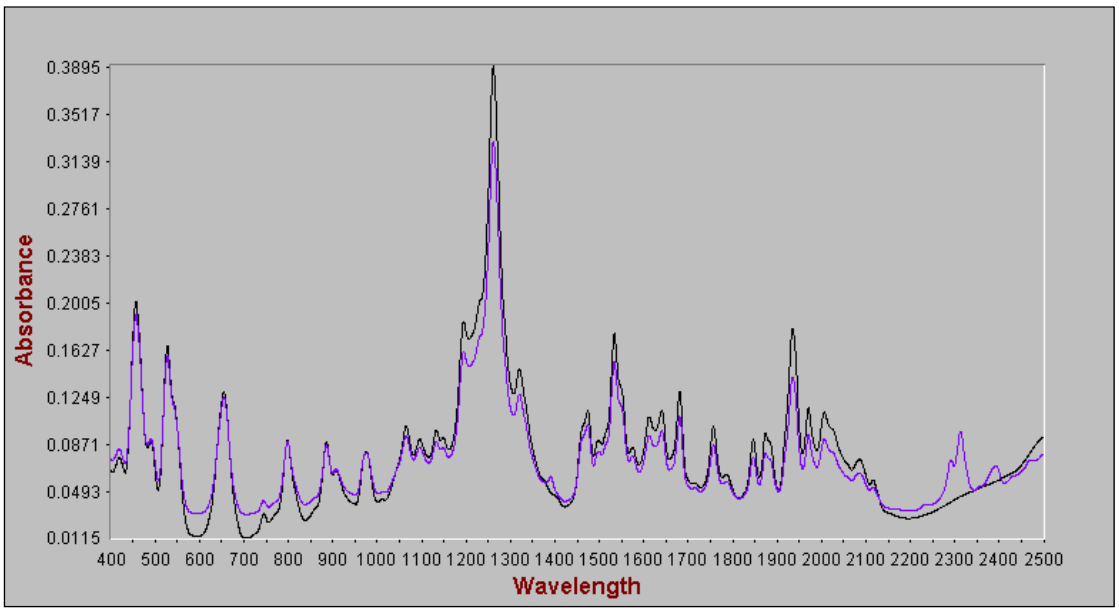
The wavelength standard used is directly traceable to NIST SRM-1920a, through direct comparison on the Metrohm master reflectance instrument, and in chemical formulation. In addition to the prescribed formulation, one additional ingredient is added, in a small amount, to provide peaks beyond those normally found in SRM-1920a. This material has very sharp bands, which are found to be stable and repeatable.

Spectra of each are as described:

The darker spectrum, which has no discernible peaks beyond about 2150nm, is SRM-1920a.

The FOSS WSR Wavelength Standard is the lighter spectrum, and has clear peaks visible at above 2200nm. These additional peaks are used to set the wavelength scale of the instrument to aid in instrument matching. This is one important step in calibration transfer.

The Foss WSR Wavelength Standard exhibits slightly different absorbance and baseline levels, due to the reflectivity of the added ingredient. The peaks, however, are in the same wavelength positions, and are similar in shape to SRM-1920a.



In Instrument Calibration, the XDS instrument is set to nominals which provide excellent instrument transfer, and which meet published NIST wavelengths when tested in Wavelength Certification. Peaks for the additional ingredient are set to peak nominals determined by measurement on several different types of research instrumentation.

In Wavelength Certification, the NIST-stated uncertainty of 1.0nm is applied. Tighter tolerances are not appropriate, unless NIST revises the stated uncertainty of SRM-1920a at some point in the future.

NOTE: The response of the WSR Wavelength Standard may vary slightly with temperature. This is typically in the range a few hundredths of a nanometer for small temperature variations. While this effect is small, it may cause some variation when running Wavelength Certification.

We suggest that the WSR standard be stored in the standards box, rather than inside the instrument. When running a test with the WSR, place it in the instrument as directed, then take it out and store it in the box, to keep the temperature as consistent as possible. Temperature inside the instrument may be as much as ten degrees (F) higher, and may cause slight wavelength drift, enough to cause slight wavelength errors when measuring wavelength response.

7.1.4 IPV Setup (Instrument Performance Verification)

IPV Setup is provided as a method to record initial instrument response to calibrated photometric reflectance standards. This is normally performed upon initial installation, immediately after Instrument Performance Certification (IPC), when a lamp has been changed, or when standards have been re-certified.

When the standards are scanned during IPV Setup, a file is generated, and is stored in the Vision directory. This file has the same format as the standards file, but a "V" is placed into the fourth character of the file name. This indicates that it is a "verification" file. For example, if the standards set has the serial number RSS10301, the IPV Setup file is named RSSV10301.

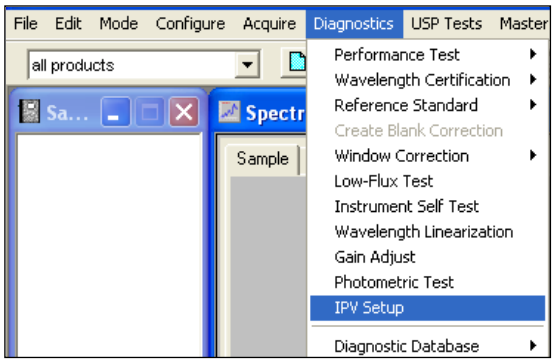
With the IPV Setup file stored, the user can run Photometric Test to check the repeatability of instrument performance. This is detailed later in this manual, in "Evaluation Diagnostics." Photometric Test compares the current performance of the instrument to the file stored during IPV Setup, and reports differences. If the instrument differences exceed established tolerance limits, the test reports that, so corrective action may be initiated.

It is important that IPV Setup, and later Photometric Test, both be run with the same options selected under Configure, Options. That is, if the IPV Setup file is acquired with Reference Standardization switched on, then Photometric Test should be performed using the same settings. The System Manager should pay particular attention to this. If options are not consistently applied, there will be a bias in the results of Photometric Test. The bias may be enough to cause test failure, depending upon selections.

Because the XDS is a sensitive instrument, it can detect differences in temperature of the standards, and results may be affected slightly. To minimize this effect, be sure the standards are at a stable temperature before use.

IPV Setup is run as follows:

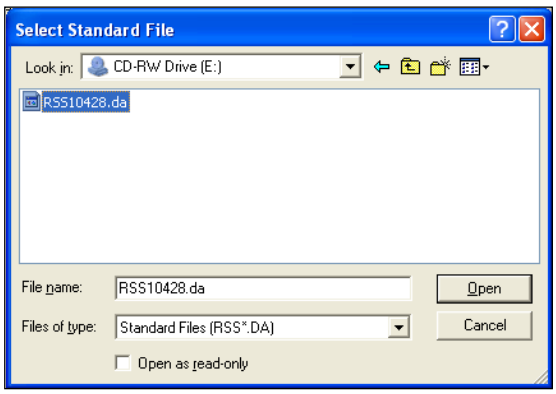
Select IPV Setup from the Diagnostics menu.



Vision requests a “Standard File”. This is provided on a mini-CD, packed in the wooden box with the standards.

Insert this mini-CD into the appropriate drive, select that drive in the dialog box, and click on the RSS1xxxx.da file as shown. (The serial number will be different, of course.) Click “Open”.

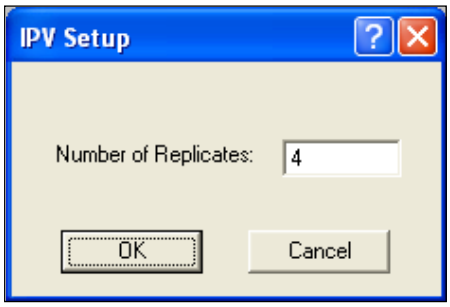
The standard file is “NSAS File” format, which refers to an older software package. This format is used where it aids in file transfer.



For Number of Replicates, enter “4”. Because the Smart Probe has a small sampling window, the replicates assure better consistency.

Click on “OK”.

Vision will begin to take an instrument reference scan, if the instrument is operating in Reference Standardized mode. The red progress bar at the bottom of the screen indicates status.



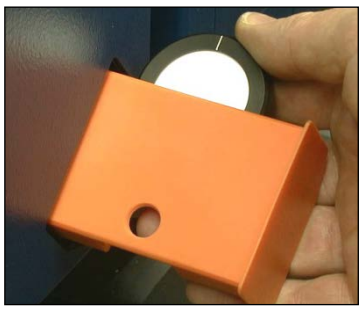
Vision will begin to take an instrument reference scan, if the instrument is operating in Reference Standardized mode. The red progress bar at the bottom of the screen indicates status.

If operating in Reference Standardized mode, Vision requests the 99% standard from the set.

(If not operating in Reference Standardized mode, Vision requests the 80% standard, which is used in place of the internal instrument reference.)

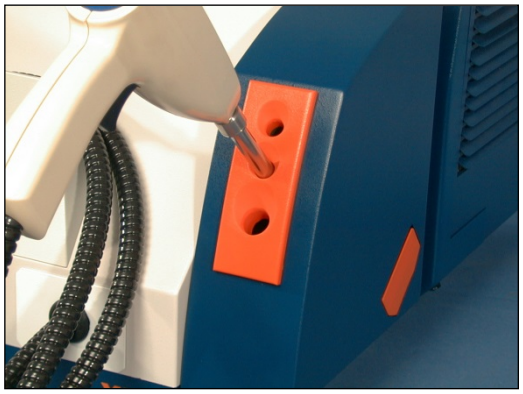
Place the 99% standard in the sample drawer as shown. Note the position, so that it can be rotated as directed.

The standards should have 4 scribed lines to help set the rotational position.



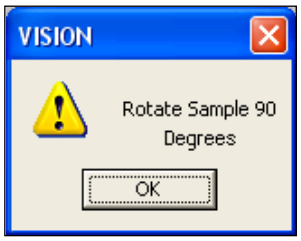
Gently place the probe into the middle position when prompted by Vision.

Close the drawer first, then gently lower the Smart Probe, into light contact with the standard window.



Vision requests rotations for replicates.

Lift the Smart Probe slightly, then rotate the standard 90 degrees each time, and continue.



Select the requested standard from the set. Labels on the back identify each standard.

Note the mini-CD that contains the "Standards File". This file is used during IPV Setup, but not during Photometric Test.

Photometric Test uses the file that is created during IPV Setup. The newly-created file is stored in the Vision directory.

7.2 Evaluation Diagnostics

Evaluation Diagnostics are used to verify that the instrument is operating within allowable parameters. These tests should be run approximately once per week.

This information is meant to guide the user through the tests in an expeditious manner. A more complete description of these tests is given in the Vision Manual, in the Diagnostics section. A discussion of the theory and interpretation of results is provided in the Vision manual.

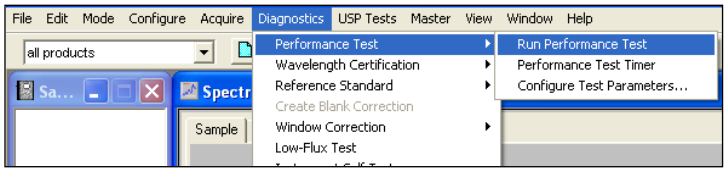
7.2.1 Performance Test

Performance Test is a comprehensive test of instrumental performance, and is the final assurance that the instrument is ready to run samples. The key items verified during this test are:

- Instrument Noise in each of four wavelength regions
- Internal Wavelength Performance (wavelength positions on non-traceable, internal reference materials.)
- Internal Wavelength Precision (Repeatability)
- NIR Gain
- Visible Gain

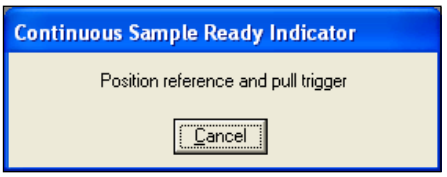
The test is initiated as follows:

Select Performance Test from the Diagnostics menu. Click on Run Performance Test.

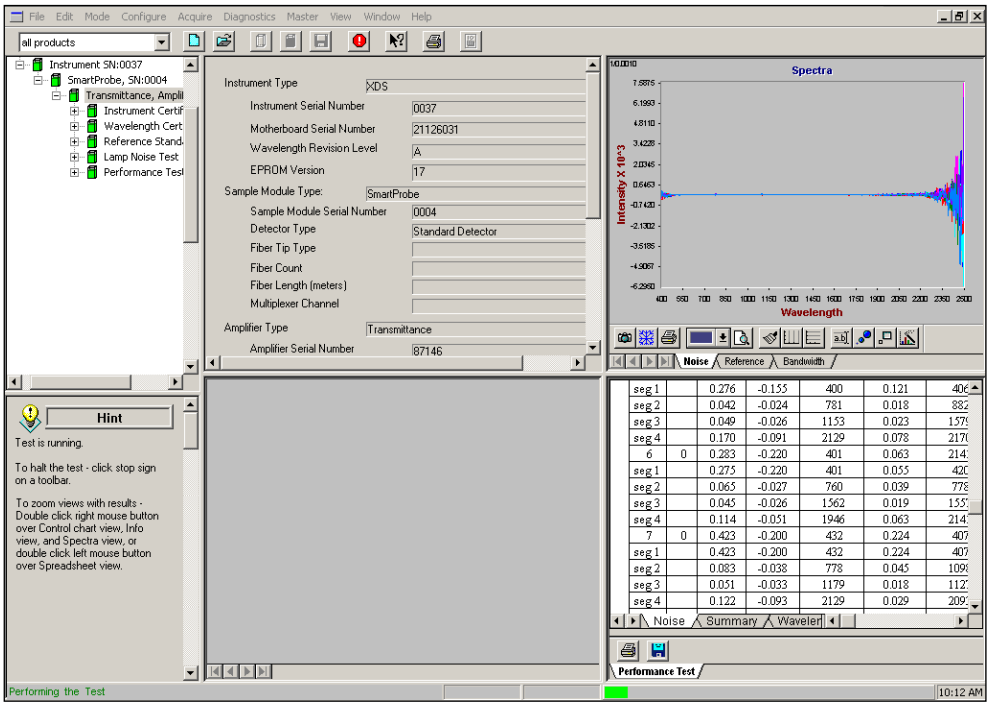


Verify that the Smart Probe is inserted into the Reference position as shown.

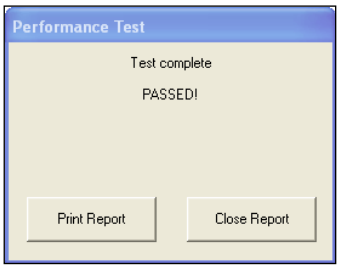
When prompted, squeeze the trigger on the Smart Probe handle to start Performance Test.



As the test runs, a screen like that shown below is displayed. Upon test completion, a message box is displayed to indicate test completion and status.

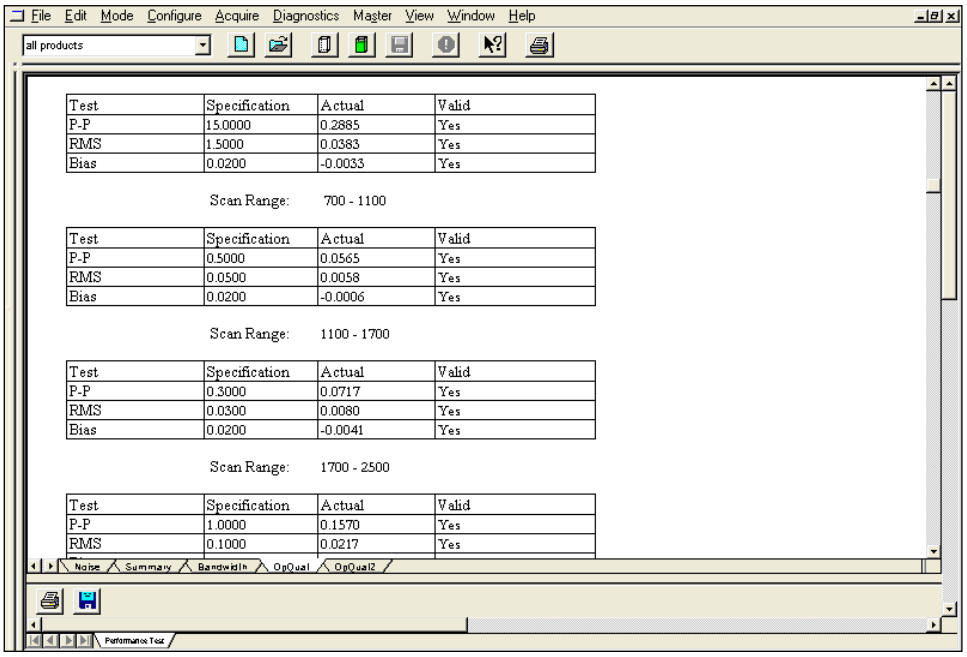


At the end of Performance Test, all measured values are compared with acceptance criteria stored in Vision. If all results meet acceptance criteria, the test is successful and this dialog box is displayed.



Before clicking "Close All Reports", the user is directed to the tabular display.

To view the tabular display of results, place cursor over the tabular display and double-click twice. Vision enlarges the tabular portion of the screen. Now click on the OpQual tab, near the bottom of the screen. Noise summary results are shown.



Noise Summary displayed using the OpQual tab.

The OpQual tab brings up the display shown. This shows results of the Noise Test for each of the four wavelength regions. These regions are:

- 400-700nm
- 700-1100nm
- 1100-1700nm
- 1700-2500nm

For each region, results are given for

- Peak-to-Peak Noise (P-P)
- Root-Mean-Square Noise (RMS)
- Bias (A measure of baseline energy changes)

Each of these parameters is described in more detail in the Diagnostics Section of the Vision Manual. If the test is reported as "Passed" the user may proceed with sample analysis.

The XDS instrument contains internal wavelength reference materials, which are used as a means to maintain monochromator wavelength measurement. These internal wavelength materials are protected and are moved by software command, transparent to the user. When Performance Test is run, the relative wavelength positions and repeatability of these wavelength materials are monitored and reported.

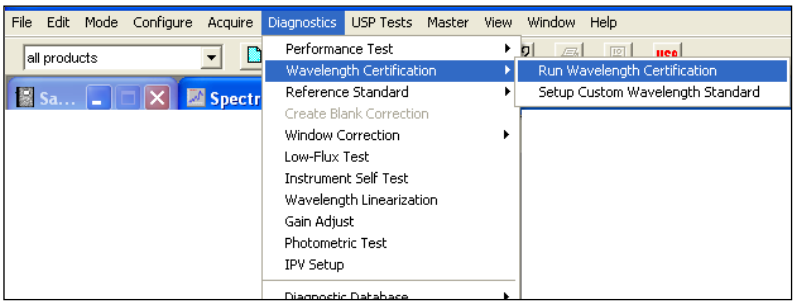
Note that these internal wavelength materials need not be precisely on the assigned nominals. These nominals are arbitrary. The internal wavelength materials are a method to assure stable readings on the external wavelength standard, measured at the sample plane.

Increasing noise in Performance Test may indicate a gradually failing lamp. Performance Test is very sensitive to any optical changes in the instrument, including noise caused by lamp issues. Be sure the instrument is warmed up, then run the test again. If it continues to fail, consider a lamp change to remedy the problem.

7.2.2 Wavelength Certification

Wavelength Certification is used to confirm the peak positions of the instrument to a defined, external wavelength standard.

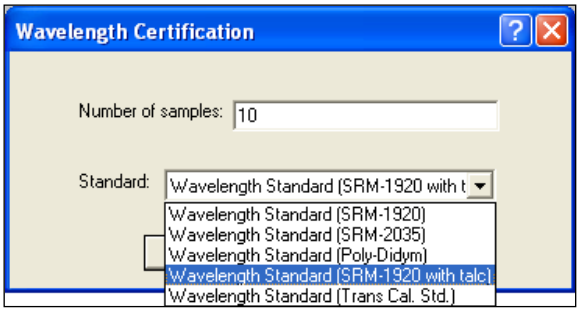
Click on Diagnostics, Wavelength Certification, Run Wavelength Certification.



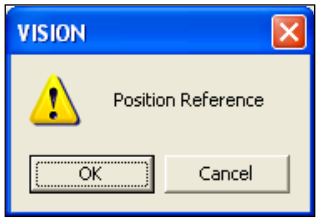
The Number of Samples should be 10, as shown. The wavelength standard for reflectance is the SRM-1920 plus talc. This is the same cell used for Instrument Calibration.

Current versions of Vision default to this selection, for this instrument. Please verify that it is correct.

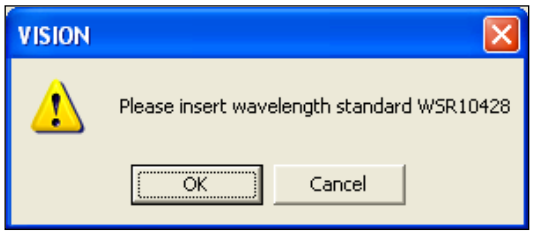
Click "OK" when ready.



Vision requests that the user place the instrument reference into position. This is automatic with the Smart Probe Analyzer; no response is required.



When the reference scan is finished, insert the wavelength standard as directed.



Always place the standard into the instrument in the same orientation. In this case, the user has marked the standard for repeatable positioning.

Click "OK" when ready.

When the test is complete, a spectrum of the standard is shown in the upper right quadrant of the screen. A tabular report is shown in the lower right quadrant, giving each peak, its nominal position and its measured position. The difference from nominal, and the repeatability of wavelength peak positions are calculated.

Instrument Wavelength Certification

Sensor Model:	XDS	Serial Num:	0037	EPROM:	17
Sample Module:	SmartProbe	Serial Num:	4	Detector:	Transmittance
Date:	7/26/2002	Time:	9:03:43		
Standard Set:	RSS10300	Type:	Reflectance	Standard ID:	WSR10300

Author/Operator: FOSS Default User

Nominal Peak Positions (nm):

655.10 886.50 975.50 1261.80 1681.40 1935.50

Measured Peak Positions

Scan	Peak 1 (nm)	Peak 2 (nm)	Peak 3 (nm)	Peak 4 (nm)	Peak 5 (nm)	Peak 6 (nm)		
1	655.08	885.92	975.22	1261.66	1680.65	1935.33		
2	655.08	885.92	975.22	1261.66	1680.65	1935.35		
3	655.08	885.92	975.22	1261.66	1680.65	1935.35		
4	655.08	885.92	975.22	1261.66	1680.65	1935.34		
5	655.08	885.92	975.22	1261.66	1680.65	1935.34		
6	655.08	885.92	975.22	1261.66	1680.65	1935.34		
7	655.08	885.92	975.22	1261.66	1680.65	1935.34		
8	655.09	885.92	975.22	1261.66	1680.65	1935.35		
9	655.08	885.92	975.22	1261.66	1680.65	1935.35		
10	655.09	885.92	975.22	1261.66	1680.65	1935.35		

Summary of Accuracy

	Peak 1 (nm)	Peak 2 (nm)	Peak 3 (nm)	Peak 4 (nm)	Peak 5 (nm)	Peak 6 (nm)		
Average	655.08	885.92	975.22	1261.66	1680.65	1935.35		
Tolerance	+ 1.00/- 1.00	+ 1.00/- 1.00	+ 1.00/- 1.00	+ 1.00/- 1.00	+ 1.00/- 1.00	+ 1.00/- 1.00		
Delta	-0.02	-0.58	-0.28	-0.14	-0.75	-0.15		
S/D	0.003	0.001	0.001	0.002	0.001	0.006		
Max	655.09	885.92	975.22	1261.66	1680.65	1935.35		
Min	655.08	885.92	975.22	1261.66	1680.65	1935.33		
Max-Min	0.0084	0.0037	0.0025	0.0052	0.0039	0.0210		
Result	Pass	Pass	Pass	Pass	Pass	Pass		

Instrument Profile Positions (nm):

527.5 798.0 1064.5 1193.0 1681.5 1970.0 1642.0

Instrument Profile and Bandwidth nominals (nm):

12.55 14.50 14.80 13.65 11.45 9.95 8.78

Measured Instrument Profiles and bandwidth

Scan								Bandwidth
1	12.55	14.46	14.82	13.65	11.46	9.95		8.77
2	12.55	14.47	14.82	13.64	11.45	9.95		8.77
3	12.55	14.46	14.82	13.63	11.46	9.94		8.83
4	12.55	14.45	14.82	13.65	11.48	9.94		8.84
5	12.55	14.47	14.82	13.64	11.46	9.99		8.85
6	12.55	14.46	14.82	13.62	11.48	9.99		8.78
7	12.56	14.47	14.82	13.61	11.49	9.96		8.81
8	12.55	14.46	14.83	13.63	11.46	9.96		8.78
9	12.55	14.46	14.82	13.63	11.47	9.97		8.78
10	12.55	14.46	14.82	13.64	11.47	9.98		8.78

Summary of Accuracy

	Position1 (nm)	Position2 (nm)	Position3 (nm)	Position4 (nm)	Position5 (nm)	Position6 (nm)		Bandwidth
Average	12.55	14.46	14.82	13.64	11.47	9.96		8.80
Tolerance	+ 0.10/- 0.10	+ 0.10/- 0.10	+ 0.10/- 0.10	+ 0.10/- 0.10	+ 0.10/- 0.10	+ 0.10/- 0.10		+ 0.15/- 0.15
Delta	0.00	-0.04	0.02	-0.01	0.02	0.01		0.02
S/D	0.003	0.007	0.003	0.012	0.011	0.022		0.030
Max	12.56	14.47	14.83	13.65	11.49	9.99		8.85
Min	12.55	14.45	14.82	13.61	11.45	9.94		8.77
Max-Min	0.0098	0.0223	0.0100	0.0396	0.0333	0.0475		0.0853
Result	Pass	Pass	Pass	Pass	Pass	Pass		Pass

If the test has passed, the user should click the cursor on the tabular display, go to the File menu, and click Print to keep a copy of the report. It is saved in the Diagnostic Database for future recall. See the Vision manual for a full explanation.

Wavelength Certification also tests certain peaks in the wavelength standard. These peaks are used to set the "Instrument Wavelength Profile," and one peak is used for bandwidth calculation.

The wavelengths used for the instrument wavelength profile are well-defined, stable peaks in the wavelength standard. These are the same peaks used during Instrument Calibration. Wavelength Certification is a verification that the peaks are in correct positions, and that the peak positions are

consistent over time. Note that both tests use the wavelength standard at the sample plane, where actual sample measurement is done.

Performance Test measures peak positions of internal reference materials, and these positions are used as a method to maintain wavelength measurement at the sample plane, using the external standard. As discussed under Performance Test, the internal reference materials are not traceable, and are only used as an internal method of maintaining correct wavelength measurement on the external wavelength standard.

In the event that the instrument passes Wavelength Certification on the NIST-defined peaks, but fails on the FOSS-defined (tighter) peaks, this message may be displayed.

This is not a failure; it is a message that the Instrument Calibration peaks have drifted slightly and should be re-run. This could be due to ambient temperature issues with the WSR, or drift due to other causes.

Re-run Instrument Calibration, then run Wavelength Certification again to verify that it has passed.

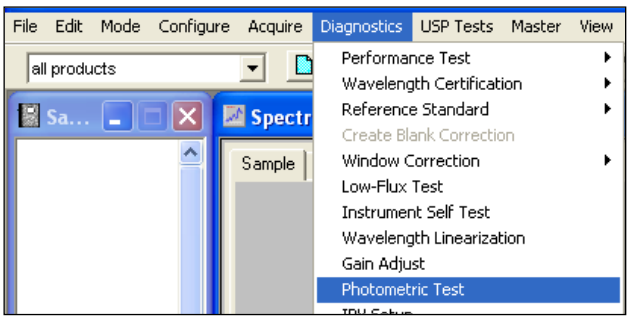
7.2.3 Photometric Test

Photometric Test provides a method to verify ongoing photometric performance of the instrument. This is a requirement for pharmaceutical users. Test results are stored in the Diagnostic Database, and may be accessed at any time. Control charts are plotted (after several tests have been stored) to provide an ongoing record of performance.

The test uses the same standards used in IPV Setup. Photometric Test compares current spectra of each standard to those stored during IPV setup. If any differences exceed normal tolerance values, the instrument can be assumed to have changed in some manner, and may need service.

Because the calibrated photometric standards are the link to previous photometric performance, the standards should always be stored in their wooden box, and protected from fingerprints, dropping, or other damage. If any cup is opened, dropped, or otherwise altered, Photometric Test results may fail.

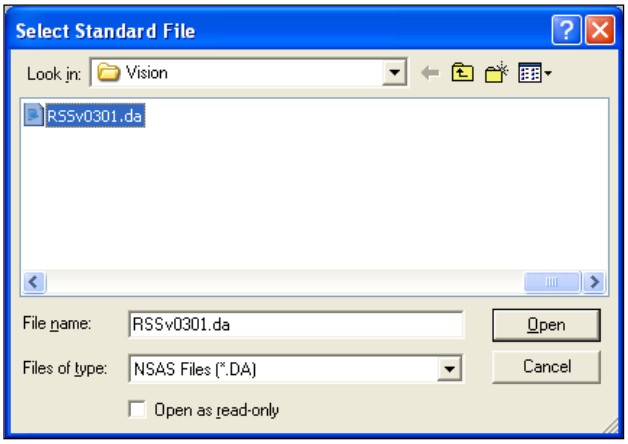
To run Photometric Test, select it from the Diagnostics menu.



Vision requests a “Standard File”. For Photometric Test, use the RSSVxxxx.da file stored in the Vision directory. This file was created during IPV Setup. Current photometric readings will be compared to that initial file.

Click on the RSSVxxxx.da file as shown. (The serial number will be different, of course.) Click “Open”.

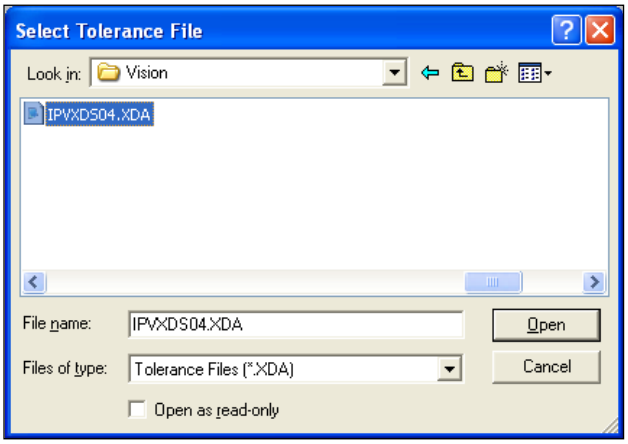
Do not use the file on the standards mini-CD for Photometric Test, as it will cause Vision to return an error message.



Vision requests a tolerance file. The tolerance file was loaded in the C:\Vision directory, and is an “XDA” formatted file.

Select this file and click “Open”.

Note that tolerance files for Photometric Test are numbered. The digits “04” refer to the fourth version of this tolerance file. When new XDS instruments are added, the tolerance file number is incremented. Tolerances for a given instrument do not change within the file, however. (The file IPVXDS01.xda will provide the same test tolerances as IPVXDS04.xda.)



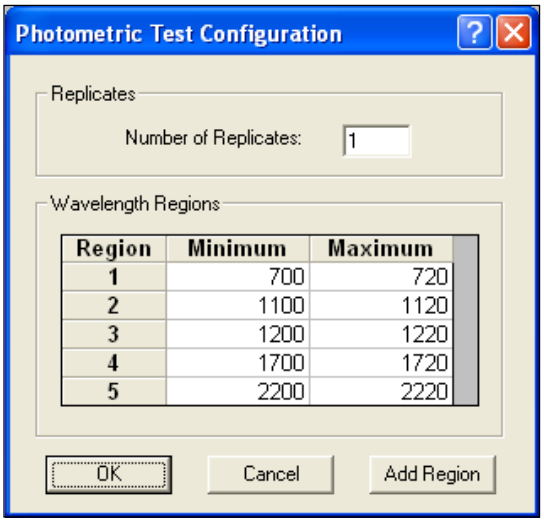
Vision displays the wavelength regions for test.

For reflectance instruments, these regions give a good overall picture of instrument performance and repeatability. The wavelength areas are chosen in flat parts of the standards spectra for stability.

For Number of Replicates, Enter “4” as in IPV Setup.

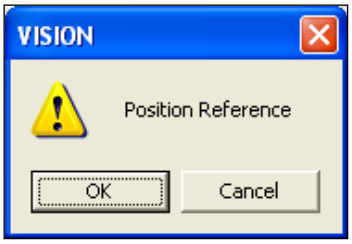
Click on “OK”.

Vision will begin to take an instrument reference scan, if the instrument is operating in Reference Standardized mode. The red progress bar at the bottom of the screen indicates status.



Vision requests “Position Reference”.

The Smart Probe should be placed in the top opening, gently resting against the Spectralon instrument reference.



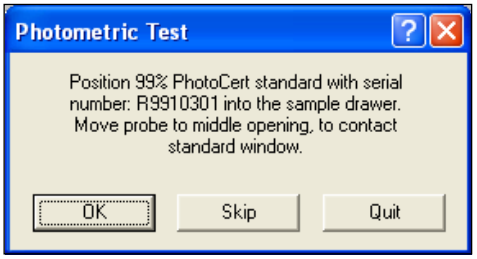
This shows the correct Reference position for the XDS Smart Probe.

(Do not press hard, as this may change the reflectivity of the material.)

NOTE: The instrument reference is Spectralon®, which is a PTFE material. This instrument is designed for use with raw powders, with no sample window. The Spectralon does not have a quartz window over it, because a window would change the reflectivity. Therefore it is very important not to allow dirt to contact the reference.

If operating in Reference Standardized mode, Vision requests the 99% standard from the set.

(If not operating in Reference Standardized mode, Vision requests the 80% standard, which is used in place of the internal instrument reference.)

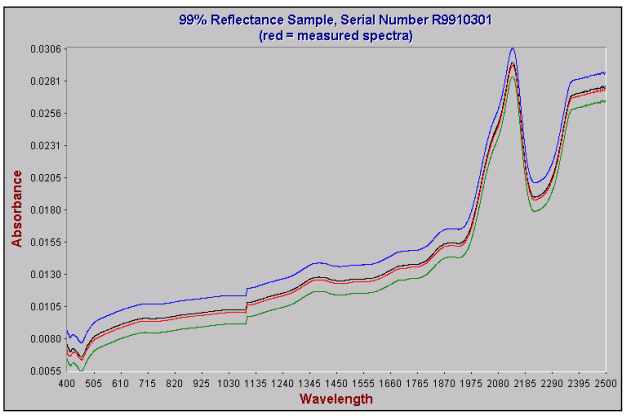


Select the requested standard from the set. Labels on the back of each standard identify the reflectance value. Place in drawer (note rotational position) and close drawer fully.

Move the Smart Probe from the top opening (reference) to the middle opening. Gently lower it to contact the standard. Do not push on the glass, as this may scratch the quartz window.

The standards used for this test are R99, R40, R20, R10, and R02.

When the 99% standard has been scanned, the result will be plotted as shown.



In this picture, the upper and lower spectra are tolerances from the initial IPV Setup spectrum. The IPV Setup spectrum is the dark spectrum in the middle, displayed in black on screen.

The lighter spectrum in the middle (red on screen) is the current spectrum. It should be within the upper (blue) and lower (green) spectra as shown.

After each standard is run, Vision plots the comparison for each wavelength area as shown. Tolerances are automatically applied, and a "Pass" or "Fail" indication is given.

Continue to follow the on-screen prompts for each standard. Vision requests the 40%, 20%, 10%, and 2% standards.

When Vision has completed the test, the tabulated results may be printed. They are also stored in the Diagnostic Database for later recall.

When the test is complete, click "Close Window".

Instrument Photometric Verification Report

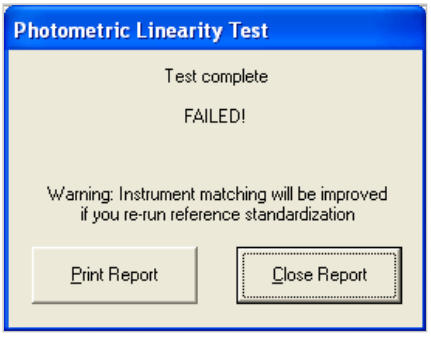
Sensor Model:	XDS	Serial Num:	0004	EPROM:	14
Sample Module:	Smart Probe	Serial Num:	0006	Detector:	Reflectance
Date:	8/21/2002	Time:	18:46:10		
Standard Set:	RSSv0301	Type:	Reflectance	Tolerance File:	IpvXDS01

Author/Operator: FOSS Default User

Photometric Standards		Reference:	R8010301		
Standard Serial Number	Test Segment NM	Reference Value Log(1/R)	Tolerance Log(1/R)	Average Bias Log(1/R)	Result
R9910301	700- 720	0.00958	0.0011 / -0.0011	-0.00022	Pass
R9910301	1100-1120	0.01083	0.0011 / -0.0011	-0.00020	Pass
R9910301	1200-1220	0.01138	0.0011 / -0.0011	-0.00021	Pass
R9910301	1700-1720	0.01368	0.0011 / -0.0011	-0.00021	Pass
R9910301	2200-2220	0.01938	0.0011 / -0.0011	-0.00021	Pass
R4010301	700- 720	0.34547	0.0027 / -0.0027	-0.00016	Pass
R4010301	1100-1120	0.34616	0.0027 / -0.0027	-0.00012	Pass
R4010301	1200-1220	0.34755	0.0027 / -0.0027	-0.00012	Pass
R4010301	1700-1720	0.35387	0.0027 / -0.0027	-0.00011	Pass
R4010301	2200-2220	0.36075	0.0027 / -0.0027	-0.00013	Pass
R2010301	700- 720	0.64969	0.0054 / -0.0054	-0.00027	Pass
R2010301	1100-1120	0.66026	0.0054 / -0.0054	-0.00018	Pass
R2010301	1200-1220	0.66384	0.0054 / -0.0054	-0.00019	Pass
R2010301	1700-1720	0.67856	0.0054 / -0.0054	-0.00015	Pass
R2010301	2200-2220	0.69277	0.0054 / -0.0054	-0.00008	Pass
R1010301	700- 720	0.99808	0.0107 / -0.0107	0.00009	Pass
R1010301	1100-1120	1.03670	0.0107 / -0.0107	0.00025	Pass
R1010301	1200-1220	1.04488	0.0107 / -0.0107	0.00026	Pass
R1010301	1700-1720	1.07448	0.0107 / -0.0107	0.00032	Pass
R1010301	2200-2220	1.09735	0.0107 / -0.0107	0.00051	Pass
R0210301	700- 720	1.93734	0.0511 / -0.0511	0.00215	Pass
R0210301	1100-1120	1.90512	0.0511 / -0.0511	0.00293	Pass
R0210301	1200-1220	1.88976	0.0511 / -0.0511	0.00282	Pass
R0210301	1700-1720	1.79654	0.0511 / -0.0511	0.00172	Pass
R0210301	2200-2220	1.71508	0.0511 / -0.0511	0.00111	Pass

A tab on this display also shows results of the USP Photometric Linearity Test, formerly suggested by USP. Note that this test was dropped by USP as of December 1, 2008. Vision contains the tab for those users who are required to run it by internal procedures. Metrohm does not suggest or require the USP test.

In some cases, Photometric Test may fail and display the message shown. This indicates that the photometric scale of the instrument has shifted outside of Photometric Test tolerances.



- Failure may be due to several possible things:
- The instrument Spectralon reference has become soiled, and changed reflectivity, usually indicated by the red spectrum being below the green spectral limit,
- The instrument needs to have Reference Standardization re-run,
- The probe window may be soiled,
- Some combination of the above.

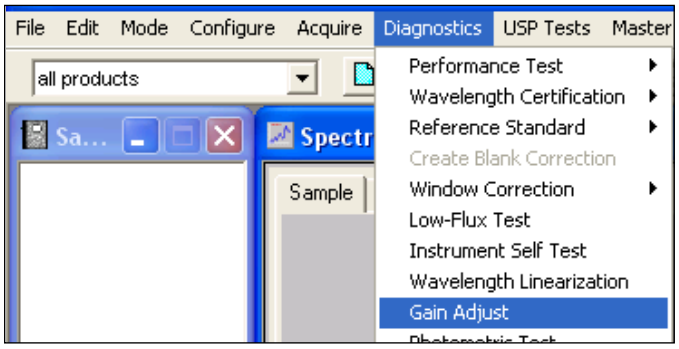
The user should verify proper test methods, cleanliness, and Reference Standardization. If the Spectralon reference is quite dirty, it may require replacement and subsequent re-Reference Standardization.

7.2.4 Gain Test

The Gain Adjust feature can be a useful diagnostic tool, though it is not required for normal operation. Technically, gain is never adjusted on the XDS Smart Probe Analyzer. The name of the test comes from a capability required with older systems. With XDS, this program reports gain information for the NIR and visible regions.

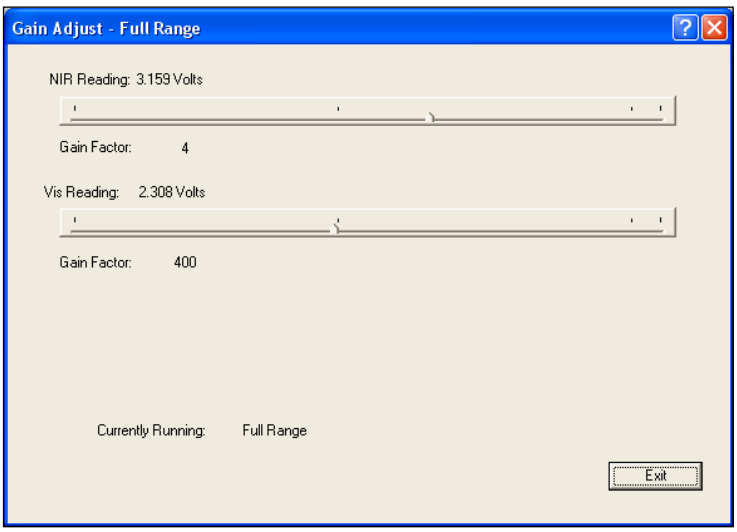
To start Gain Adjust, click on Diagnostics, then Gain Adjust.

The instrument is connected and in communication for this to function.



This view shows a fairly typical Smart Probe Analyzer instrument. The gain program requires that the Interactance Reflectance probe be in the reference position (top opening). Vision takes gain readings for both NIR and Visible regions.

Gain Factor is a measure of signal amplification. In the NIR region (1100-2500nm) it occurs in steps of 1, 2, 4, 10, 20, 40 and 80. In the Visible region (400-1100nm) the gains range from 1 to 80,000.



Gain Adjust can be helpful when troubleshooting an instrument. For example, a gain of 80 in NIR and 80,000 in Visible is a sign that the lamp is burned out, or some other sort of failure. Note that the gain factors are reported in Performance Test, and can be called up from the Diagnostic Database. This permits the user to see if the gain factor has changed significantly over time.

7.2.5 Low Flux Test

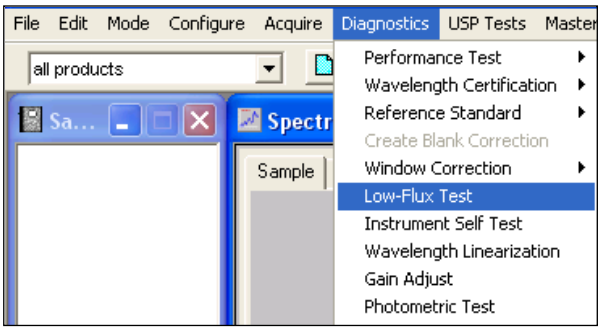
Low Flux Test is included for users who must run this test in support of regulatory requirements.

Low Flux Test uses a nominal 10% reflectance standard in the sample position. A noise test is run using this standard. Because the reflectivity is less than the instrument standard, the test is considered a good method for testing instrument noise in the range of reflectivity of many common sample absorptions.

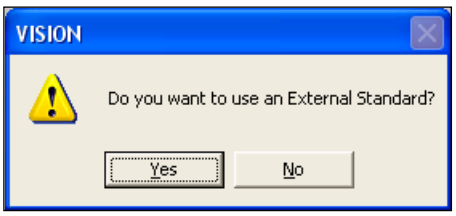
The XDS instrument has an internal 10% neutral density filter (transmittance) screen, triggered by software, which can be used in place of an external 10% reflectance standard. This screen gives equivalent results during the Low Flux Test, and minimizes the possibility of operator error in placing the standard.

To initiate the Low Flux Test, follow this sequence:

From the Diagnostics menu bar, select Low Flux Test.



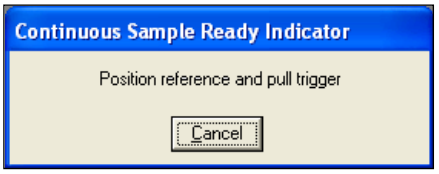
Vision asks if the user wishes to use an external sample (standard) for the test. Click “Yes” to use an external 10% reflectance standard.



XC-1010 Reflectance Standards contain a 10% reflectance standard (R101xxxx) which may be used for this test.

The 10% Reflectance Standard must be placed into the drawer on the side of the instrument, with the reflectance probe inserted into the middle opening for this test.

Follow instructions for trigger pull.



If the user clicks “No” to the external standard, then Vision will automatically trigger the 10% internal screen for this test. In this case, the reflectance probe should be in the upper opening, against the internal instrument standard.

Vision runs the Low Flux Test, which takes about 10 minutes. At the end, the results are displayed. A typical test result is shown:

Low Flux Noise Test

Date: 7/12/2002 Time: 15:42:44
Instrument Model: NIRSystems XDS
Serial number: 0004
EPROM Version: 164
Wavelength Rev: A
Motherboard S/N: 00000130
Sampling System: Smart Probe Module

Noise Test (mA) Scan Range: 400 - 2500
seg 1 400 - 700
seg 2 700 - 1100
seg 3 1100 - 1700
seg 4 1700 - 2500

Scan	EOC	P-P	Minimum	Wavelength	Maximum	Wavelength	Bias	RMS	Gain
1	0	0.823	-0.338	2396	0.484	400	-0.011	0.055	20 | 400
seg 1		0.590	-0.106	439	0.484	400	0.023	0.068	
seg 2		0.025	-0.012	733	0.013	882	0.001	0.006	
seg 3		0.165	-0.079	1529	0.086	1108	-0.008	0.025	
seg 4		0.551	-0.338	2396	0.213	2421	-0.033	0.076	
2	0	0.674	-0.163	2478	0.512	2466	0.024	0.056	20 | 400
seg 1		0.540	-0.099	423	0.441	409	0.019	0.063	
seg 2		0.111	-0.014	896	0.097	1100	0.001	0.009	
seg 3		0.138	-0.060	1100	0.078	1546	0.013	0.025	
seg 4		0.674	-0.163	2478	0.512	2466	0.044	0.078	
3	0	0.900	-0.544	2489	0.356	2421	0.024	0.053	20 | 400
seg 1		0.369	-0.157	408	0.212	400	0.000	0.034	
seg 2		0.024	-0.007	1081	0.017	846	0.005	0.004	
seg 3		0.128	-0.029	1104	0.100	1309	0.029	0.023	
seg 4		0.900	-0.544	2489	0.356	2421	0.040	0.081	
4	0	1.421	-1.030	400	0.391	2494	-0.012	0.059	20 | 400
seg 1		1.218	-1.030	400	0.188	406	-0.026	0.082	
seg 2		0.049	-0.016	784	0.033	1100	-0.003	0.005	
seg 3		0.152	-0.108	1111	0.044	1643	-0.015	0.028	
seg 4		0.706	-0.315	2422	0.391	2494	-0.008	0.078	
5	0	1.124	-0.601	400	0.523	2496	0.033	0.063	20 | 400
seg 1		0.641	-0.601	400	0.040	455	-0.038	0.080	

Click on the tab marked "Summary" to see the summarized results as compared to acceptance specifications. Vision reports a pass or fail based upon successful test completion.

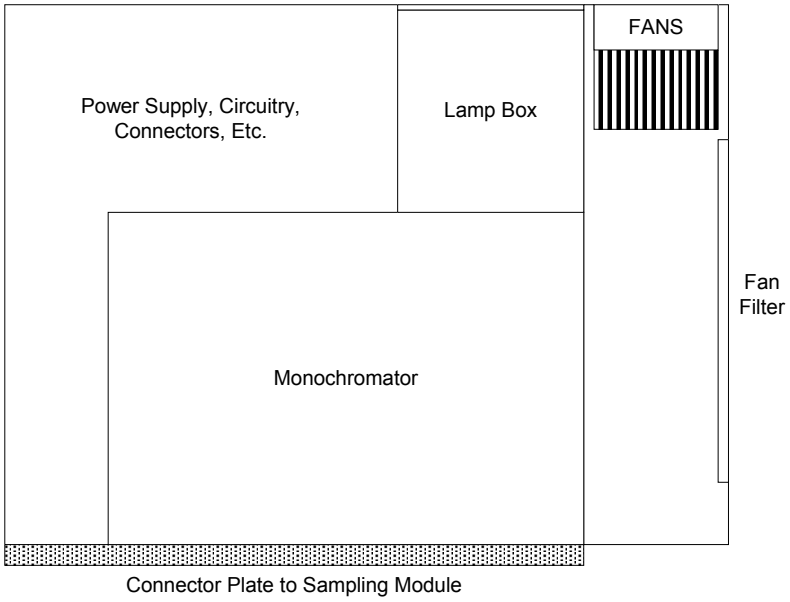
Results are stored in the Diagnostic Database for later recall. The user may print results, or click "Close" to complete the test.

8 Instrument Maintenance

Instrument maintenance is quite simple on the XDS Smart Probe Analyzer. The optical enclosure is sealed to prevent contamination of critical parts, which keeps maintenance to a minimum. A diagram of the internal parts is shown, primarily for user information.

8.1 Overview

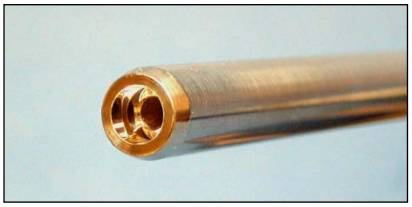
DO NOT attempt to open the optical enclosure. There are no user-serviceable parts inside. Damage is not covered under warranty.



This is a diagram of a top view of the inside of the instrument, showing the relative location of major components. Do not attempt to open the instrument enclosure unless directed to do so by trained Metrohm service personnel.

The instrument should be kept clean at all times. If it becomes dusty or dirty, wipe it with a damp, soft cloth to restore the finish.

The sampling window should be kept clean to prevent loss of signal. (This is especially important with high-absorbance samples.) Clean with a dampened, soft, non-linting tissue. Remove all surface deposits and dust.



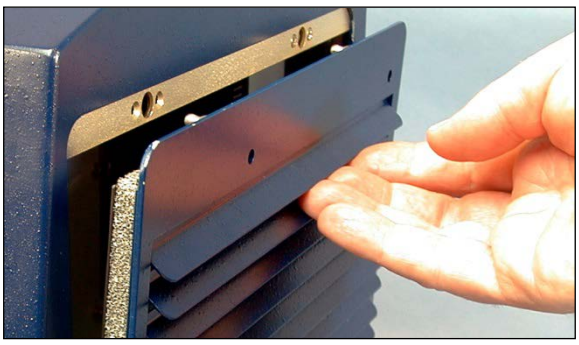
Do not scratch the Smart Probe window. Scratches will cause scattering of signal, and may affect analytical results.

Periodically verify that no equipment has been placed nearby that might produce vibration or impacts that could be transferred to the XDS Smart Probe Analyzer. Such equipment can produce spectral disturbances that are visible in diagnostics scans, and may affect analytical results.

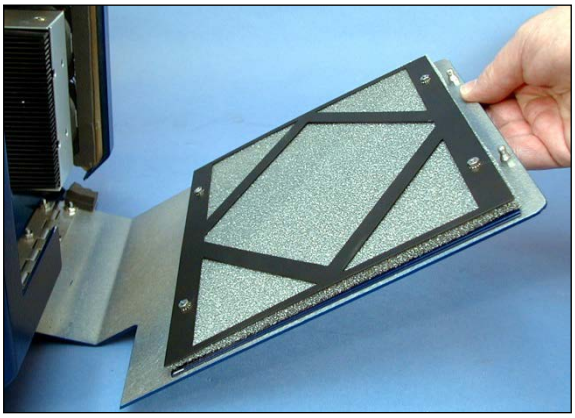
8.2 Fan Filter Replacement

The fan filter should be inspected at least monthly. (If installed in a dusty or dirty environment, it should be checked weekly or twice-weekly.) The filter is changed as follows:

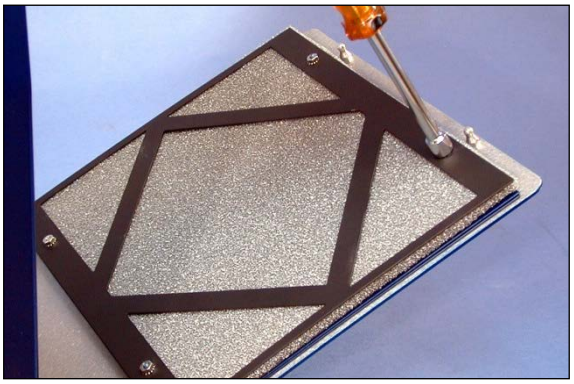
Open the door of the filter compartment. Grasp the upper fin as shown and gently pull. The snaps should release, allowing the door to open as shown.



Lower the door as shown to expose the filter material.

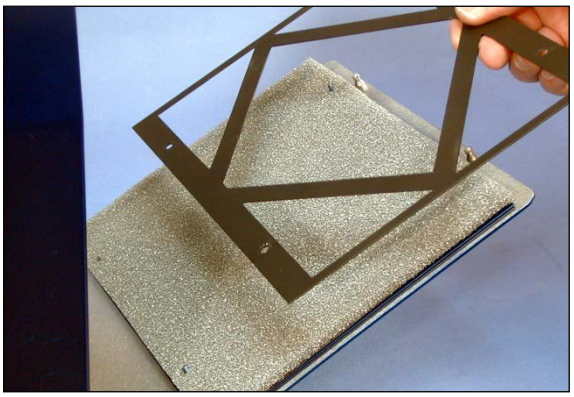


Using a 1/4" nut driver, remove the four #4-40 nuts that hold the filter frame in place.



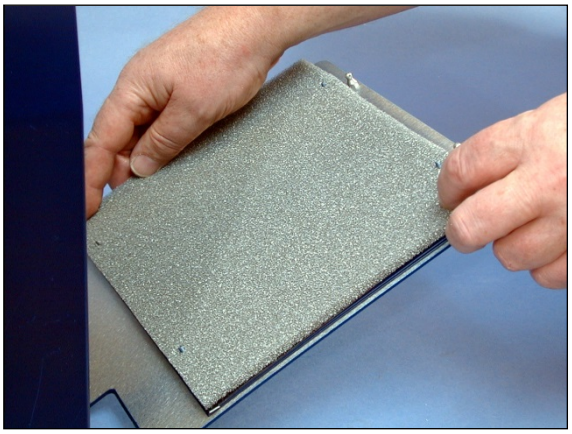
Lift the filter frame off and carefully set it aside for re-use with the new filter material.

Do not lose the nuts.

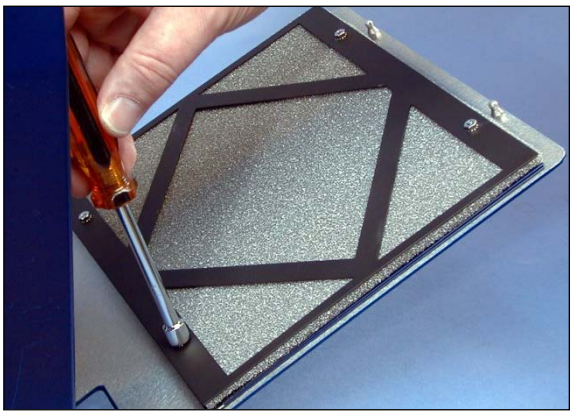


Lift the old filter material off the door screws and discard.

Install the new, clean filter material carefully over the screws as shown. The screws should penetrate the filter material fully.



Install the filter frame. Install the #4-40 nuts and tighten (hand-tight) as shown.



Gently close the instrument door.

Order new filter material from your Metrohm authorized distributor immediately.

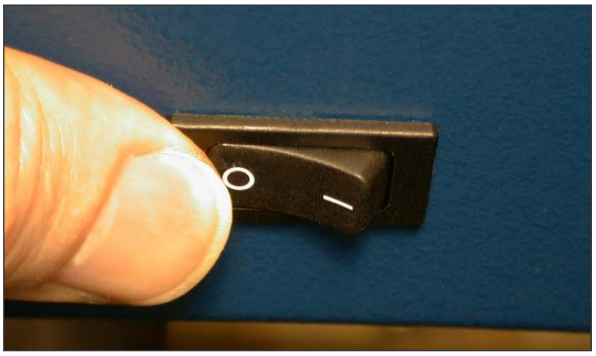
8.3 Lamp Replacement

The lamp may need to be changed after several thousand hours of use. Generally the instrument will exhibit high noise during Performance Test, or when wavelength precision (repeatability) has begun to rise from established values. Follow this procedure:

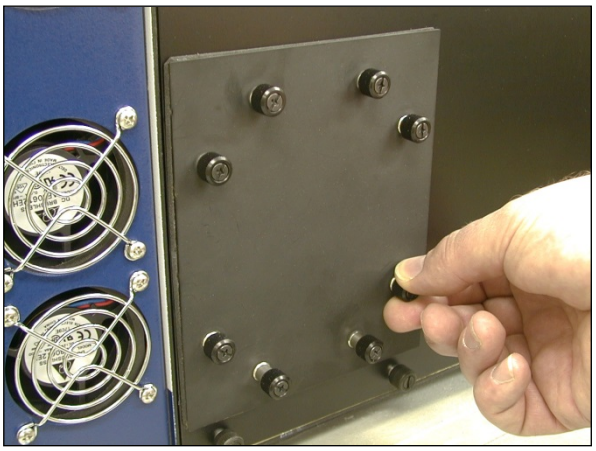
1. Turn off Instrument power and unplug AC power cord from AC receptacle or supply.

   The "O" position is OFF.

   Allow the instrument to cool for about 15-20 minutes before attempting to change the lamp.

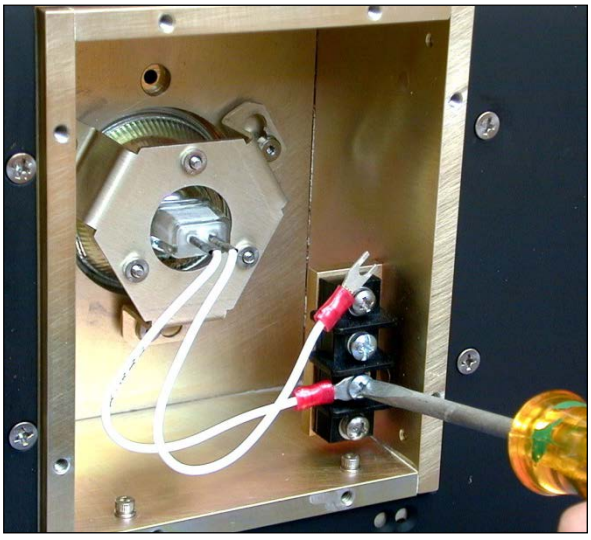


2. On the back side of the instrument, loosen the eight (8) captive thumbscrews that secure the lamp cover in place.

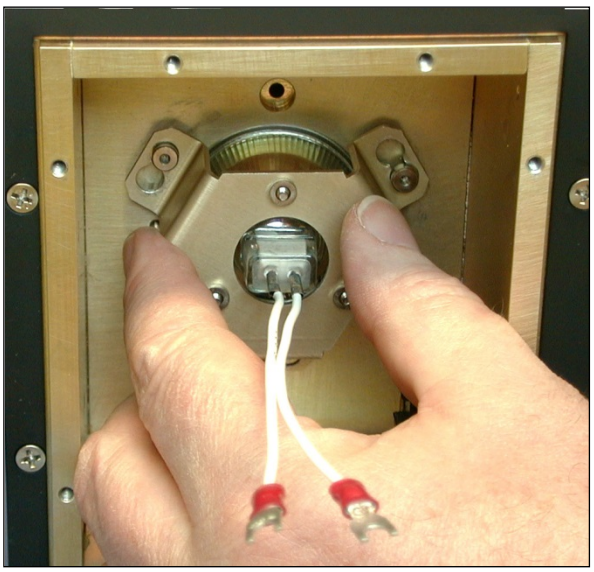


3. Loosen the screws holding the wire terminals onto the terminal strip. Do not remove these screws. One or two turns counter-clockwise is sufficient.

   Pull the wire connectors out from under the screws.

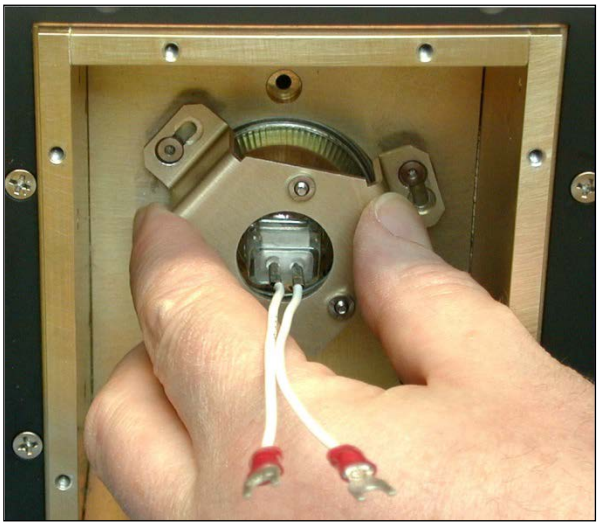


4. Grasp the lamp retaining ring as shown. Push inward slightly, and rotate clockwise (the top to the right; the bottom to the left) to release the retaining ring from the shoulder screws.



5. This photo shows the lamp retaining ring in the removal position.

   The spring will tend it push the retaining ring outwards when the shoulder screws line up with the openings. Do not drop the lamp or retaining ring.

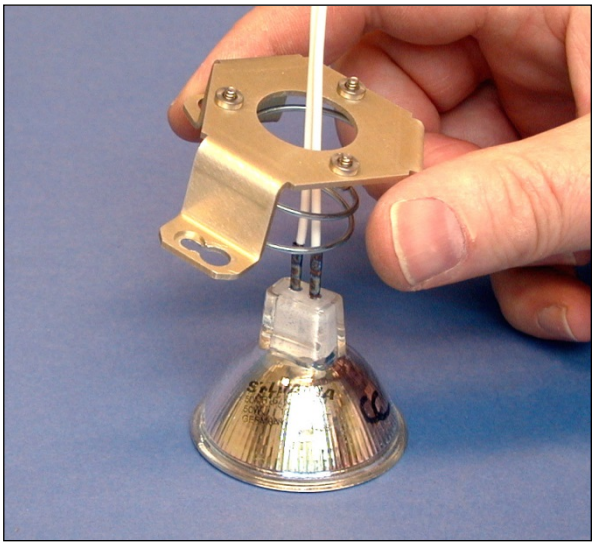


6. Pull the lamp and ring outward from the instrument as shown.

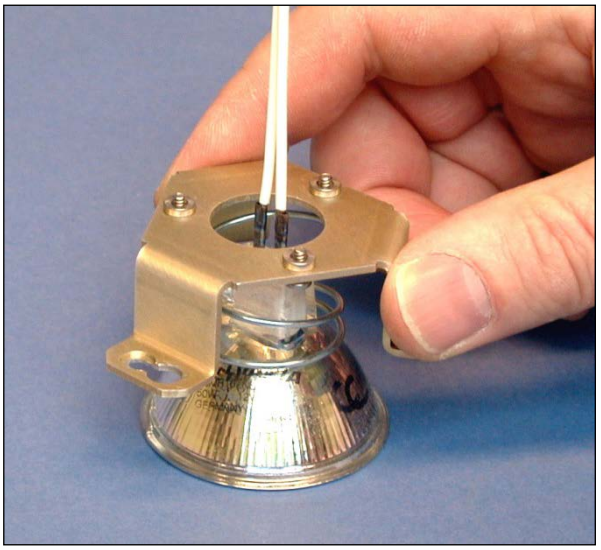
7. With the lamp wires straight, lift the lamp retaining ring upward and off the lamp as shown.

   Discard this lamp immediately. Order a new lamp from your Metrohm distributor to keep for the next lamp change.

   Always keep a spare lamp in stock, to avoid last-minute emergencies. The part number is XA-3000.

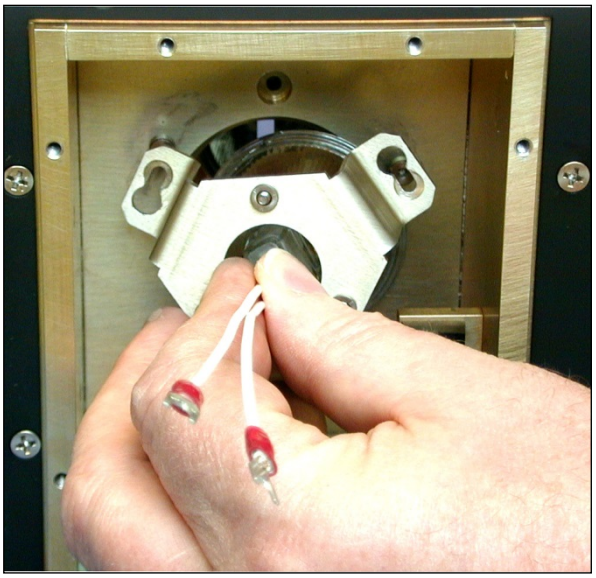


8. Unpack the new lamp from the protective box. Place the new lamp carefully onto the bench, and place the lamp retaining ring over the wires as shown.



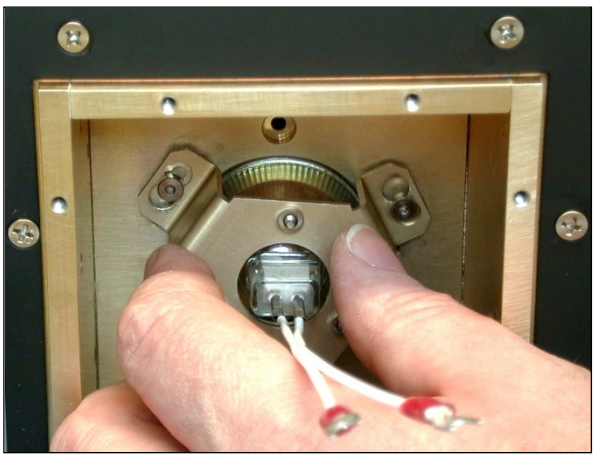
9. Gently place the lamp and retaining ring into the lamp box in the instrument.

   The outer edge of the lamp should fit into the machined inset in the lamp plate.



10. Place the lamp retaining ring over the shoulder screws, and rotate counter-clockwise (top side to left, bottom to right) to lock the lamp retaining ring into place.

    This photo shows the ring being rotated into position.

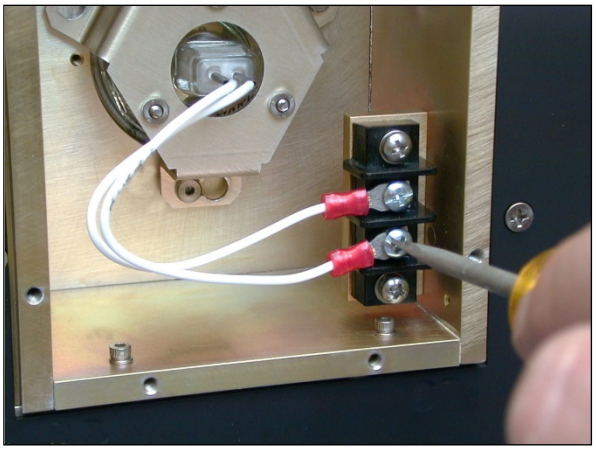


11. When the lamp is properly locked, the assembly will appear as shown. Note the shoulder screws holding the lamp retaining ring.

    The arrow must be at the top center location as shown. This is important for proper operation during Instrument Calibration.

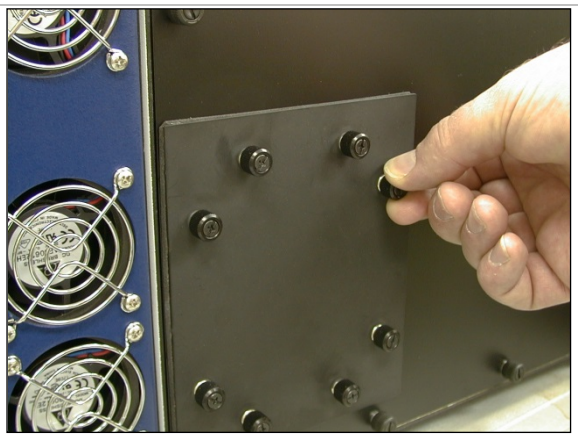
    The rectangular base of the lamp may be in any orientation. We have placed it horizontally for appearance purposes only.

12. Install the wire terminals to the terminal strip as shown. There is no polarity. Place one wire to each terminal. Tighten securely.



13. Place the lamp door over the lamp box, and tighten the eight (8) thumbscrews to hold the door in place.

    Do not use any tools to tighten the thumbscrews.



14. Plug the instrument back in to AC power. Turn the AC power switch to ON.

15. Connect to the instrument through Vision software.

    Verify that the Lamp LED (green, upper right) illuminates.

    Let the instrument warm up until the "Ready" LED (green, middle LED on lower row) illuminates to indicate that operating temperature has been reached.

16. Perform Reference Standardization (if enabled) and Instrument Calibration. Run Performance Test. If all tests pass, the instrument is ready for operation.

Always keep a spare lamp in stock, to avoid last-minute emergencies. If you do not have a spare lamp after changing this lamp, order one from your Metrohm distributor immediately.

NOTE: The lamp is a very special assembly, and should never be replaced with any substitutes. Special features of the lamp include (but are not limited to) the following:

Hand-selected for high-energy filament placement

Coated internally and externally for maximum NIR performance

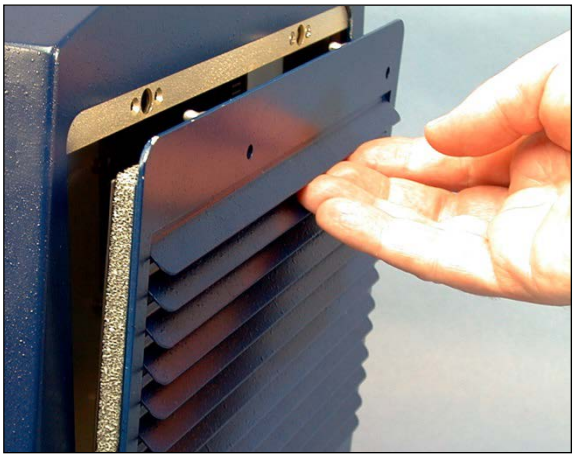
Welded wire connectors to minimize resistance and assure consistent illumination

Tested for low-noise spectroscopic performance

8.4 Fuse Replacement

Fuse Replacement is an unusual event, and usually is caused by some electrical fault. The electrical fault should always be investigated and repaired before fuse replacement. Once the fault is found and corrected, this procedure should be followed:

Turn off power and unplug the instrument from AC power. Open the side cover.



Remove the AC power plug from the AC power block of the instrument.

Use a tool (such as an Allen wrench) to pull the housing open.

There is a small slot where the tool can be inserted to pry the door gently open.

Remove old fuses from holder and discard. Install the new fuses as shown.

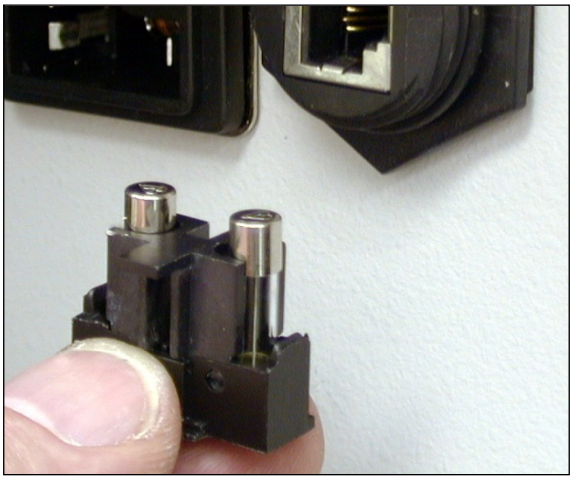
Fuse Ratings:

5A 250 VAC, 5 x 20mm

Slo-Blo

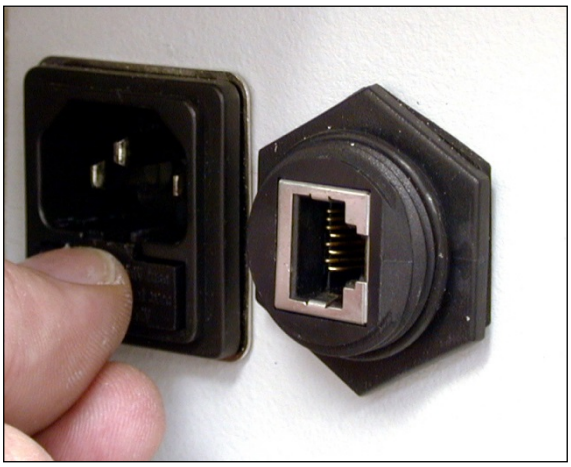
(2) Required

They clip in to the plastic holder, and should be positioned at the center of the holder.



Close the fuse door fully. Plug the AC power cord in. (Plug the RJ-45 cable in if it was removed.)

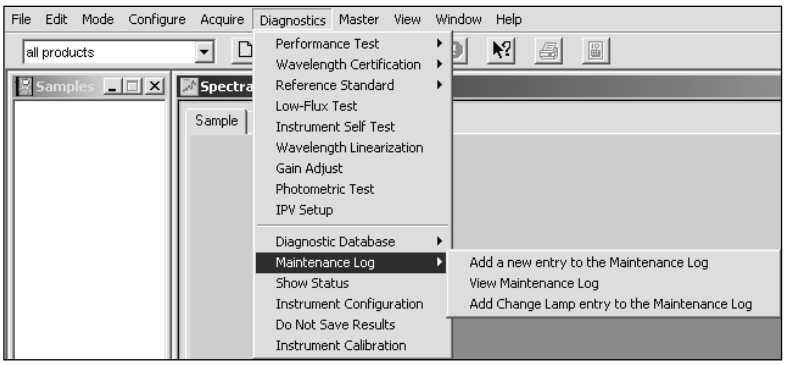
Turn on AC power and re-establish instrument communication.



8.5 Maintenance Log

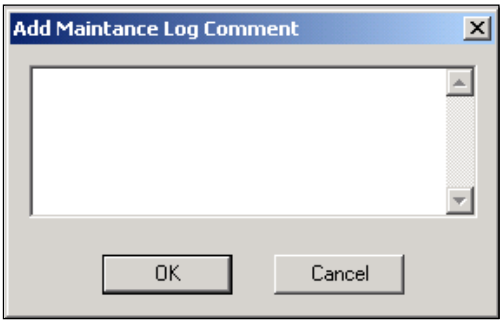
Vision provides a Maintenance Log in the Diagnostic Database to permit tracking of maintenance activity. This provides a convenient place to find information about tests, lamp changes, and user-entered comments.

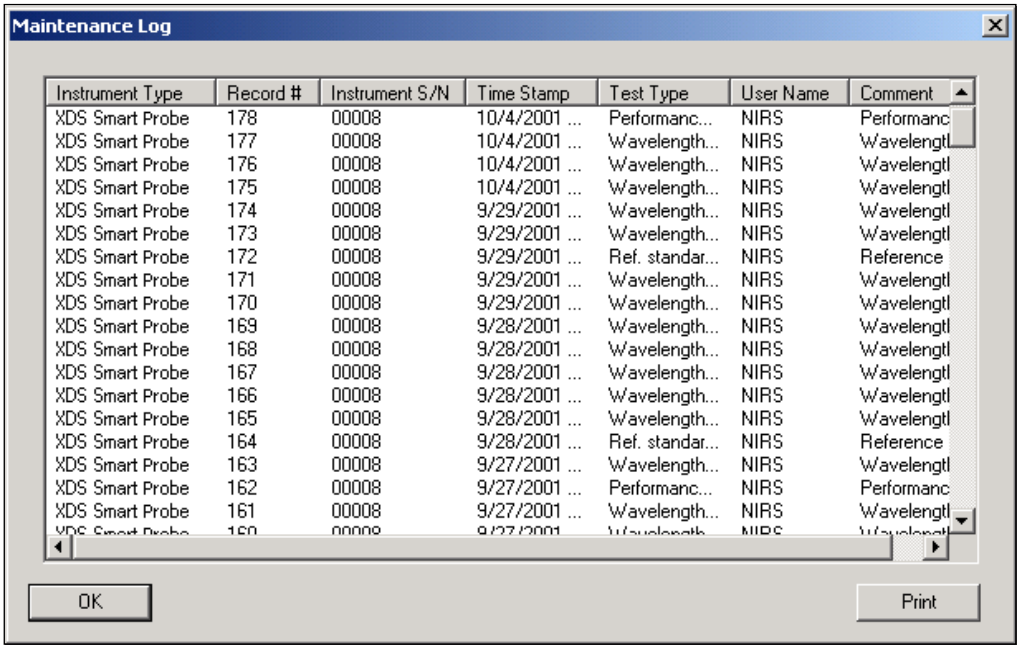
To access the Maintenance Log, click on Diagnostics, Maintenance Log, then the correct selection.



Maintenance Log tracks all instrument tests, as shown in the screen below. In addition, the user may enter text comments, which are saved as part of the log.

Enter the text, then click on OK.





This screen shows entries, sorted by record number. To re-sort by another parameter, click on the column heading.

9 Validation Tools

Validation is an overriding concern in the pharmaceutical marketplace. In the United States, manufacturers must follow Title 21 of the Code of Federal Regulations, also known as CFR 21. Each country has its own regulations or has adopted a set from another source; therefore, the requirement for validation is worldwide.

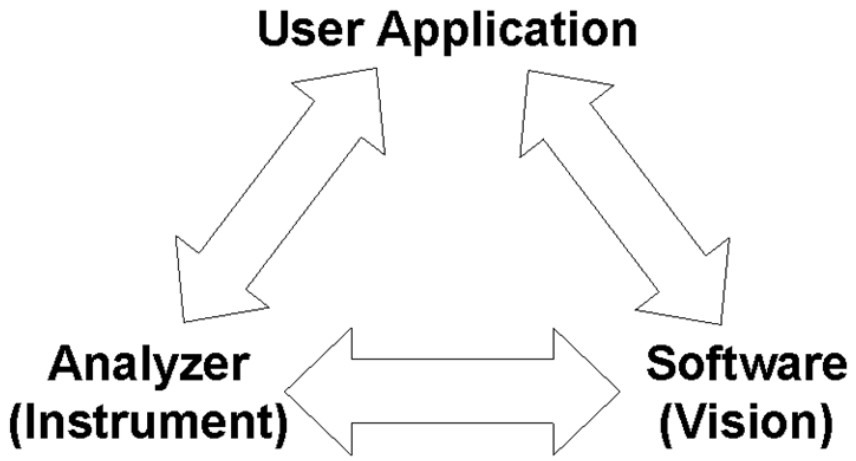
The requirements are very detailed, and will not be recounted here. For every instrument used to measure, qualify, or release materials at any stage of the pharmaceutical manufacturing process, there must be a thorough validation package to support it. This is an onerous but necessary task.

In the chemical and polymer industries there is a heightened awareness of Q9000, often referred to as ISO9000. This regulation is similar in scope and intent to CFR 21, but has not been uniformly enforced with analytical instrumentation. There are many reasons for this, including assessor familiarity with NIR instrumentation. However, the enforcement is beginning to be applied more commonly.

In summary, validation is or will be the concern of every analytical instrument user, sooner or later. Validation is generally broken down into three categories:

- **Hardware** (Analyzer and sampling accessories)
- **Software** (Vision Spectral Analysis Software)
- **User Application** (Customer samples, limits detection, range of calibration set, calibration precision, and other factors.)

Users have requested specific tools and techniques to achieve validation. Metrohm has provided the following tools to assist and expedite the task. Each item is described.



Successful validation must include all three elements: The Analyzer, Vision Software, and the User Application.

9.1 Hardware Validation Tools

Factory Instrument Test Guide and Results

Every Metrohm instrument is factory-tested, using the same battery of tests provided for ongoing

customer testing. Included with your instrument are results from Performance Test, Photometric Test, and Wavelength Certification. These factory tests are performed under controlled, ideal conditions, and serve as an important baseline for all subsequent testing.

Test results are included for each module or configuration ordered. These test results are included in an informative brochure that explains the tests, what they measure, and how they relate to instrument performance. The Instrument Test Guide and the user instrument test results form the first part of the instrument log recommended by regulatory bodies.

9.1.1 Installation and Operating Qualification Documents

Laboratory-based Industrial instruments come with Installation Qualification (IQ) and Operating Qualification (OQ) Documents included as part of the Vision® Spectral Analysis Software package. These documents are presented in checklist fashion, to guide the user through each step of instrument set-up and qualification.

9.1.2 NIRStandards® for Instrument Performance Verification

Various regulatory bodies recommend regular testing of NIR reflectance instrumentation to verify continuing stability of photometric and wavelength response. These reflectance standards include Metrohm IPV® Software. It records "first use" instrument response, and permits regular verification of subsequent response to the original measurements.

NIRStandards are designed for easy use with all Metrohm reflectance instruments used in the pharmaceutical industry, and most Metrohm instruments used in other industries. They are calibrated on our Master Instrument for use with the Instrument Performance Certification Program.

9.1.3 Instrument Performance Certification

The United States Code of Federal Regulations, Title 21, recommends regular instrument testing, as well as periodic testing of a deeper nature to assure instrument response and reliability. Instrument Performance Certification (IPC) is offered to meet these requirements. It is performed every six (6) months, on site, by a certified technician. Some key program features:

- Measures instrument response using standards calibrated to Metrohm Master Instrument
  Assures consistent response between instruments of identical design.
- Performed by factory-trained service personnel
  Periodic maintenance is performed on-site to assure consistent ongoing performance.
- Serves as Installation Qualification and Operating Qualification for new instrument installations
  Meets CFR 21 requirements for instrument qualification.

- Independent assessment, not performed by instrument user
  Meets independent test requirements of CFR 21 and most auditing methods.
- Full documentation of all tests, adjustments and findings
  Serves as Instrument Log, with valuable records of ongoing performance.

9.1.4 Metrohm Master Instrument Program

In support of our worldwide base of instruments, Metrohm maintains a Near-Infrared Master Reflectance Instrument. This instrument is calibrated with vaulted standards, using several different (yet complementary) techniques to assure consistency and accurate response. Full records of testing and calibration are logged and maintained on an ongoing basis.

By maintaining a Master Instrument, Metrohm is able to track long-term response of field instruments, verify ongoing operational improvements to manufactured systems, and provide yearly re-calibration of NIRStandards used in Instrument Performance Certification.

This long-term, stable Master Instrument assures our users of consistent measurement methodology between instruments, and for a given instrument over time.

9.2 Software Validation Tools

9.2.1 Installation and Operating Qualification Documents

Software Installation Qualification (IQ) and Operating Qualification (OQ) Documents are included as part of the Vision® Spectral Analysis Software package. These documents are presented in checklist fashion, guiding the user through each step in setting up and qualifying the instrument.

The software OQ is a pre-built project model, using actual spectra. It is imported for quantitative and qualitative analysis, and output results are calculated. The calculated results are matched with known results from a validated system, to verify installation and operation on the user computer system.

9.2.2 Vision Certificate of Validation

A Certificate of Validation is included with every Vision Software Package. This states that Vision is validated and is signed by an officer of FOSS.

9.2.3 Vision Validation Document Package

This Compact Disc (CD) contains full documentation of the Vision Spectral Analysis Software design process. This CD is available for purchase through your Metrohm Distributor.

Documentation begins with the original Functional Requirement Specification, Software Requirement Specification, Architecture Documents, Code Reviews, Test Results, Acceptance Tests, and other important information.

Running to several thousand pages, this CD will satisfy your Internal Audit Staff and external regulators that Vision Software was designed from the first moment to be a fully code-validated,

tested product for use in the pharmaceutical industry. Documents were selected (from the enormous quantity of records in our logs) based upon the patterns set during the many customer audits of our software development process. Documents may be printed using Adobe Acrobat®, which is included on the CD for download.

This CD is the most comprehensive, informative record of software validation anywhere in the industry today. FOSS is also willing to host software audits (Upon agreed notice and terms) to those customers wishing deeper information.

FOSS will host software audits by the FDA or other recognized regulatory bodies upon customer request. Normal audit terms and scheduling policies will apply. Escrow agreements for source code are available.

9.2.4 21 CFR Part 11 Compliance

Vision meets the strict requirements of 21 CFR (Code of Federal Regulations) Part 11, covering Electronic Records and Signatures. Key compliance features include:

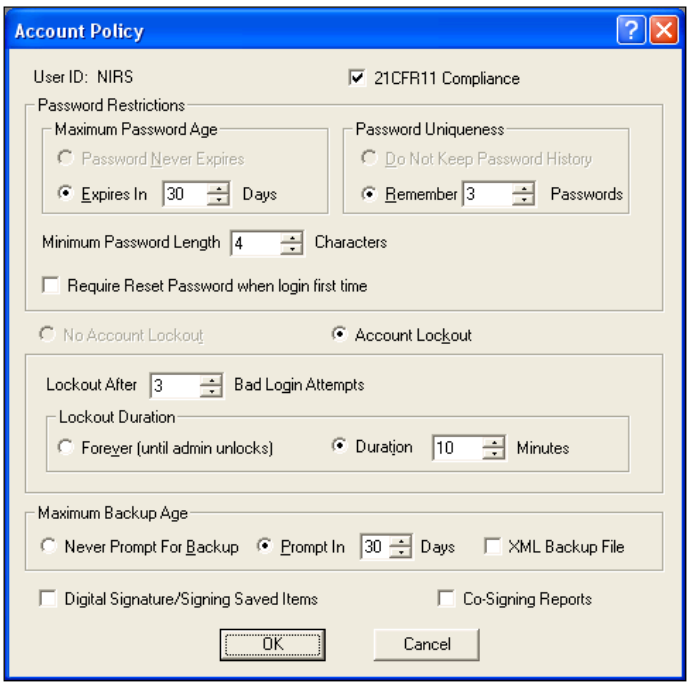
- Validation of system software and instrument connection
- Blocking of invalid or altered records
- Generates accurate and complete copies of records in human readable and electronic form
- Records protected and retrievable throughout their retention period, archive and backup functions provided
- Limited system access to authorized individuals with unique User ID and Password
- Secure, computer-generated, time stamped audit trail that independently records the date and time of operator entries/actions that create, modify, or delete electronic records
- Record changes shall not obscure previously recorded information
- Operational system checks to enforce permitted sequencing of steps and events, as appropriate
- Use of device (e.g. terminal) checks to determine the validity of the source of data input or operational instruction

This screen shows many of the setup items to be set by the System Manager for 21 CFR Part 11 Compliance.

To access this screen, click on Configure, Account Policy.

A 21 CFR Verification Document is available for users who wish to verify key features. This document is included with Vision installation materials, on the same CD, starting with Vision 3.50, SP1.

Please contact your local distributor for information, or if you cannot locate this document.



10 Safety and Electrical Certification

The XDS Smart Probe Analyzer and all associated components have been tested for CE (Communite European) certification. An independent, accredited laboratory is used for this testing.

CE certification is a comprehensive set of requirements that encompass user safety, immunity from electrical interference, and low radiated electrical emissions. The requirements overlap with UL and CSA requirements in nearly all areas. As a rule, CE certification is recognized by most countries as acceptable for installation.

All major electrical components used in the instrument meet UL, CE, CSA, or TUV certification. Usage is as defined by the manufacturer, to assure safe performance. Wire sizes, colors, and terminations meet CE requirements. All connectors meet CE safety standards. Shielding is provided to avoid user contact with hazardous voltages during use of the instrument. Additional safeguards have been taken to avoid defeating these shields and interlocks.

Circuit boards and electrical signal lines have been design for minimum radiated electrical emissions, and are designed for superior immunity to outside electrical interference.

All sources of thermal energy have been evaluated. The instrument design is meant to dissipate any thermal energy. A circuit interlock is provided as a backup to primary thermal controls. The instrument has been tested at maximum operating temperature, with all functions active, as a means of verifying thermal dissipation under worst-case conditions.

Normal care should be exercised by the user to avoid conditions which might be deemed hazardous. This includes, but is not limited to:

- Spillage of liquids on and around the instrument,
- Use of the instrument at temperature higher than those listed (95 deg. F, 35 deg. C,
- Operation at voltages or supply frequencies outside those listed (100-240VAC, 50-60Hz),
- Unauthorized use of accessories that might alter or circumvent electrical safeguards,
- Blockage or disabling of cooling fans or air filter, causing elevated temperatures.

This instrument is to be used solely for the purpose intended by the manufacturer. All other uses are strictly prohibited.

There are no user-serviceable parts inside the instrument. Do not open or attempt to service the instrument in any way, other than those operations described in this manual.

In the unlikely event of problems, contact Metrohm service or your local authorized Distributor.

This equipment is to be used only for the purpose specified. If used for any other purpose, the protection provided by the equipment may be impaired.

11 Troubleshooting

The XDS Smart Probe Analyzer is a dependable, trouble-free instrument, designed for many years of service in your laboratory. In spite of the rugged design, problems may arise that require attention. This guide is intended as a means of diagnosing minor problems.

There are no user-serviceable parts inside the instrument enclosure.

Because of this design, we emphasize that under no circumstances should the user attempt to open the instrument cabinet and service any part. The components are may be damaged or misaligned by handling. Hazardous voltages may be present even with power removed from the system.

Any diagnosis of internal function should be performed using software diagnostics, not by internal inspection.

For minor problems, this guide should be consulted. While some recommendations are quite basic, some of the suggestions may be helpful in avoiding oversights or problems. Follow the recommendations and eliminate any possible causes listed. Recommendations are listed in logical order of occurrence wherever possible. When all recommendations have been checked, and if the instrument is not operating, please contact Metrohm Service or your local authorized distributor if the problem is not solved.

	Observed Problem	**Recommendations**
1	Instrument does not "power up".	1. Verify that AC power cord is plugged in to AC power source. 2. Verify that the AC power cord is plugged into the instrument AC power block. 3. Verify that the AC power switch is turned on. 4. Verify that AC power is available at the AC power source, using an AC voltmeter. 5. Check for blown fuses. If fuses are blown, investigate and repair the cause, then replace fuses.

2	No communication between Vision and the instrument.	1. Verify that the RJ-45 cable is plugged in at both the instrument and at the network wall jack. 2. Verify that the RJ-45 cable is plugged in at both the computer and at the network wall jack. (**NOTE:** Direct connection is explained in section 3.0 for non-network users. This requires a special cable. 3. Verify that the instrument is powered on. (See previous Observed Problem.) 4. Verify that the network wall jack is active, and has a connection point within the internal network. 5. Verify that the instrument is Available in Configure, Input. This instrument serial number is found on the serial plate on the side of the instrument. This serial number should be visible in Vision in Configure, Input. 6. Verify that the connector ends of the RJ-45 cable are not damaged, crushed, or distorted in any way. Wires should be firmly clinched by the connectors.
3	Lamp does not come on when instrument is connected.	1. Verify communication with instrument. Click on Acquire, Disconnect, then Acquire, Connect, Select DCM to verify proper connection. If lamp does not come on, replace lamp according to instructions provided in part 8.3 of this manual. Do not attempt to replace lamp with AC power applied. 2. Lamp may be burned out. This should not occur for thousands of hours of normal use, but could be caused by jarring or other physical motion. Replace lamp. 3. Instrument thermal shutdown may have occurred due to high internal operating temperatures. Determine cause of high temperature and correct before subsequent operation.

4	Instrument fails Wavelength Linearization	1. Verify that the Interactance Reflectance probe is positioned in the top opening, against the internal reflectance reference, when the test is initiated. 2. Verify that the Wavelength Standard is properly positioned in the sample drawer when prompted by software. Be sure the Interactance Reflectance probe is positioned in the middle opening when directed to scan the WSR Wavelength Standard, and the probe is in contact with the WSR Wavelength Standard. 3. Instrumental problems – contact Metrohm service or your authorized distributor.
5	Instrument Fails Performance Test.	1. Verify that the Interactance Reflectance probe is positioned in the top opening, against the internal reflectance reference, when the test is initiated. 2. Temperature and/or humidity may be changing rapidly during the test. This can usually be observed as large spectral activity between 1300-1400nm and 1800-1900nm. If this is the case, the instrument should be tested in a more controlled setting to verify proper operation. 3. Instrument may be located on a bench with grinders, stirrers, or other laboratory equipment which produces vibration or mechanical disturbance. This shows up as spectral activity in various areas, depending upon the transmitted frequency of the motion. Turn off all equipment that might cause such disturbance. Locate it to another part of the laboratory, or place it on isolated supports away from the XDS Analyzer. 4. Increasing noise in Performance Test may indicate a gradually failing lamp. Performance Test is very sensitive to any optical changes in the instrument, including noise caused by lamp issues. Be sure the instrument is warmed up, then run the test again. If it continues to fail, consider a lamp change to remedy the problem.

6	Instrument fails Instrument Calibration.	1. Verify that the Interactance Reflectance probe is positioned in the top opening, against the internal reflectance reference, when the test is initiated. 2. Verify that the Wavelength Standard is properly positioned in the sample drawer when prompted by software. Be sure the Interactance Reflectance probe is positioned in the middle opening when directed to scan the WSR Wavelength Standard, and the probe is in contact with the WSR Wavelength Standard. Note that Reference Standardization must be performed before Instrument Calibration. Verify that this has been done, and that Reference Standardization is checked in the DCM.
7	Instrument Gain is excessively high in Gain Test.	1. NIR Gain should always be less than 100 on the reference, unless extreme lengths of fiber are used. If gain is high, verify that the Interactance Reflectance probe is positioned in the top opening, against the internal reflectance reference. 2. Verify that lamp is lit. (see above)
8	Instrument cooling fans are operating at maximum rate.	1. Verify that air intake on side is not blocked by other equipment. Leave at least 3-4 inches (76-102mm) space by intake fins for proper airflow. 2. Verify that air filter is clean. If not, clean or replace filter. If fan speeds dropped shortly after opening fan filter door, this is a sign of a blocked filter. 3. Verify that the fan exhaust area is not blocked, restricting the fan outflow. Leave at least 3-4 inches (76-102mm) space by fan exhaust for proper airflow. 4. Check ambient temperature in area where instrument is used. If temperature is near the maximum, fan flow will be high.

9	Cooling fans do not operate.	1. Instrument may not be warmed up. Fans do not operate until the instrument is near operating temperature. 2. Ambient temperature may not require fan cooling. This is common in cool environments. To check fan operation, cycle the Power Switch. Note if the fans come on for a short burst, approximately 25 seconds after power, the fans are operating properly.
10	Software "freezes" or "lockups" during use	Usually caused by use of two network cards in PC. Data collisions are causing lost communication "packets". Remove one NIC card, and use the connection method on page 4.
11	Vision reports that there is no Reference Standardization or Instrument Calibration; suggests that these tests be run.	1. Usually caused by entering the test, then canceling, which leaves the instrument with no completed test constants. Do not cancel when these tests have been initiated. 2. May be caused by use of two network cards in PC. Data collisions are causing lost communication "packets". Remove one NIC card, and use the connection method on page 4.

If these measures do not correct the problem, please contact Metrohm service or your next local distributor.

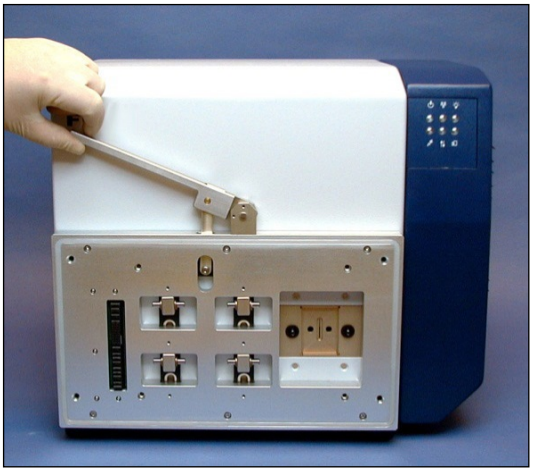
12 Lifting and transporting the Metrohm instrument:

1. Always separate the module from the monochromator (shown) before lifting or transporting the instrument.

   When the module is attached, raise this handle to separate the module from the monochromator.

   Slide the module away from the monochromator.

   The monochromator is the heavier component. Instructions follow.



2. When lifting the monochromator, place arms on either side of unit as shown.

   Lift with the knees,
   not with the back!

   Lift unit up, move to a cart, and lower gently. (Do not allow the cart to roll.)

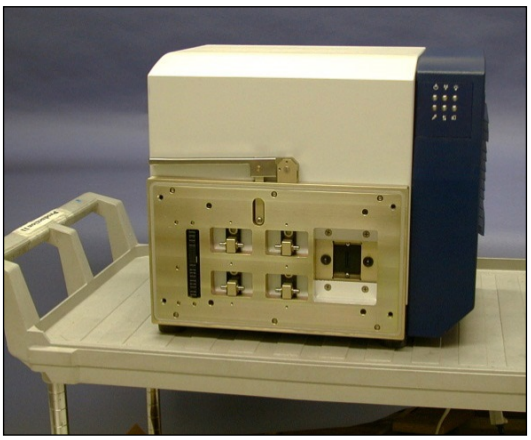
   Do not lift from below waist level, as the unit may be awkward to control.

   Ask for assistance when lifting from lower levels to bench height.

3. Always use a cart when moving the instrument from one location to another.

   The monochromator weighs 21 kilograms (46.2 lb.) and should never be carried, except when moving from a bench to a cart, or back.

   Modules typically weigh about 10.5 kilograms, or 23 pounds. Use care when handling.



Safety Notice:

This equipment is to be used only for the purpose specified. If used for any other purpose, the protection provided by the equipment may be impaired.

13 Index