NIRS
XDS RapidLiquid Analyzer

Manual
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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

Change Control (initiated with version 1.04):

<table>
<thead>
<tr>
<th>Version</th>
<th>Date</th>
<th>Summary of Changes</th>
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<tr>
<td>1.04</td>
<td>September 28, 2004</td>
<td>Address changes to reflect move to 7703 Montpelier Road, Laurel, MD 20723.</td>
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<tr>
<td>1.05</td>
<td>November 16, 2006</td>
<td>Added note about serialized XC-1300 Wavelength Calibration Standard on pages 27, 32 and 34. WST3WCAL is now serialized, and is labeled WST3W105, for example. This change permits better wavelength accuracy with the instrument, which aids in transferability between XDS Rapid Liquid Analyzers. Added last page note about closing/opening sample drawer using Vision software.</td>
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<tr>
<td>1.06</td>
<td>June 25, 2009</td>
<td>Reformatted Table of Contents. Replaced web address for FOSS NIRSystems. Inserted new information about network connection to instrument. Inserted current Configure, Options screens to manual. Moved section on opening/closing drawer to main body of manual. Removed flow-through cuvettes from manual. Added information of flow-through bulkhead fittings. Moved Wavelength Linearization ahead of Instrument Calibration. Made similar note in Lamp Replacement, that Wavelength Linearization must be run before Instrument Calibration. Clarified correct use of wavelength standards in Instrument Calibration and Wavelength Certification. These changes are fairly extensive, and are not detailed in Change Control. Please refer to the manual for a full explanation. Changes were required due to software updates over time that were designed to provide additional information to the user. Added note that Instrument Calibration is performed at room temperature. Added clarification that Blank Correction is loaded only for a given session, and does not carry over to subsequent connection sessions. Removed XC-1330 Standard Set. It is not offered at this time. Replaced photo in Lamp Replacement to show arrow on lamp reflector.</td>
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1 Introduction

Thank you for selecting the XDS™ Rapid Liquid 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.

The Rapid Liquid 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 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 Metrohm NIRSystems 6500-Series Liquid Analyzer was a versatile, proven design for NIR sampling for many years. The XDS™ Rapid Liquid Analyzer benefits from the improvements in wavelength accuracy, precision, low noise, speed, and overall performance.

A panel of 6 LED indicators provides user feedback on these functions:

<table>
<thead>
<tr>
<th>Icon</th>
<th>Status</th>
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</thead>
<tbody>
<tr>
<td><img src="image-url" alt="Power Icon" /></td>
<td>Green when power is ON</td>
</tr>
<tr>
<td><img src="image-url" alt="Network Connection Icon" /></td>
<td>Amber when connected to network or direct connection</td>
</tr>
<tr>
<td><img src="image-url" alt="Instrument Lamp Icon" /></td>
<td>Green when instrument lamp is ON</td>
</tr>
<tr>
<td><img src="image-url" alt="Scan Icon" /></td>
<td>Red when scanning reference or sample</td>
</tr>
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</table>
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-2035. The XDS Liquid Analyzer uses a three-layer calibration standard, measured against SRM-2035, to provide stable, repeatable wavelength settings. Use of this standard for Instrument Calibration enhances calibration transfer between instruments.

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 of the instrument. 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.

The Metrohm Rapid Liquid Analyzer is available with a variety of quartz cuvettes to make analysis straightforward and easy. A sealable cuvette, suitable for volatile liquids is shown.

Capped cuvettes and other styles are available, in various path lengths. Select path length based upon absorbance of the analytes of interest.

See section 5.0 for full information.
2 Site Readiness

Like most precision instruments, the Rapid Liquid 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.

Typical power consumption is 100W, 750W maximum.

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 instrument may be accessed directly through a network connection. This uses a standard RJ-45 type “patch” cable, such as CDW #074092, available from CDW Computer Centers, Inc. The instrument detects network capability and optimizes communication speed.
Alternatively, the XDS Rapid Liquid Analyzer can communicate directly with the computer by use of a UTP Crossover Cable, such as CDW #243786, supplied with unit. (gray cable)

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™ Rapid Liquid Analyzer dimensions are:

- Width: 18.0” (457 mm)
  (Leave about 6” (152mm) on the right side for ease of sample loading)
- Height: 15.0” (381 mm)
- Depth: 22.5” (572 mm) front to back

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 sample handling space.

- Monochromator Weight: 46.2 pounds (21.0 kg)
- Module Weight: 29.3 pounds (13.3 kg)

Follow lifting instructions (on last page) when moving the instrument. Avoid injury.
3 XDS 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 Direct Connection, in a 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. Metrohm 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.

### 3.3 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.

### 3.4 Flowchart Diagram of XDS Communication Protocol

```
XDS Instrument Power-up

Request Dynamic IP address from DHCP Server (or local Router)

DHCP Server Available?

YES

Dynamic IP address assigned to XDS Instrument by DHCP Server (or local Router)
(This will take about 5-10 seconds)

Patch Cable

Communication may be established between the XDS Instrument and the host computer in Vision

NO

XDS Instrument reverts to stored default IP Address 169.254.0.2
(This will take about 45 seconds)

Crossover Cable
```
3.4.1 Network Evolution Issues

This document is as correct as possible at the time of 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.

3.4.2 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.

3.5 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. If the IP Address field is empty, the user should consult “Troubleshooting Connection Problems”.

### 3.6 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. 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.

### 3.6.1 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.
3.6.2 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 Rapid Liquid 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
- Rapid Liquid Module
- 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
- XC-1300 Wavelength Calibration Standard

(Packaging may vary from that shown.)

If any items are missing, please contact Metrohm service or your next local distributor.

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 and the module serial number.

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.

The final assembly is as shown. Note that the sample drawer is closed. It will open upon power-up. Do not attempt to open the drawer manually.

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. If using a network, use a non-crossover type cable (“patch 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.

For “Direct Connection” the gray cable from the instrument plugs directly to the computer network jack. (Use the cable supplied with the instrument.)
13. 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.

14. Once the instrument has initialized, the sample drawer will quietly open as shown. Do not attempt to push or force the drawer. It is under instrument control, and is not designed for manual operation. Prepare to establish communication from Vision to the XDS instrument. This is detailed in Section 6.0, Vision Software.

This completes assembly of the XDS Rapid Liquid Analyzer.
5 Rapid Liquid Module

The XDS™ Rapid Liquid Analyzer is designed for sampling of many types of customer samples. This section describes initial calibration steps, which are detailed in section 7.1. This section gives a functional overview, so the operation can be anticipated and understood.

5.1 Wavelength Calibration of the Rapid Liquid Analyzer

The Rapid Liquid Analyzer uses an innovative method of wavelength calibration, to assure consistency between like instruments. A wavelength standard, using materials with known, stable peak positions, is used to calibrate the wavelength scale of each instrument.

This wavelength standard is calibrated on a controlled master instrument, and is characterized against SRM-2035, a National Institutes of Standards and Technology (NIST) transmission standard. This assures that wavelength registration of each Liquid Analyzer is set to known standards.

Instrument Calibration is required for transferability between instruments. It is activated under Configure, Project Options in Vision Software. This feature is fully explained in section 7.1.3.

5.2 Blank Correction

The Rapid Liquid Analyzer has the option of “Blank Correction” to provide an optimum photometric match between instruments. Vision takes a spectral scan of the sampling area (with spacer inserted), then a spectral scan of the reference path. A correction algorithm is applied to eliminate the slight difference between sample and reference.

Blank Correction must be selected in Configure, Project Options, in the screen shown at right.

A software algorithm applies the correction to each spectrum automatically. This selection feature is fully explained in section 7.1. Blank Correction is explained in section 7.1.3.
5.3 Cuvette Types

The Rapid Liquid Analyzer is preferred for samples of these types:

- Non-volatile samples, run in open or capped cuvettes.
- Volatile samples, run in sealable cuvettes.

Path length information:

As a rule, aqueous samples require path lengths of 0.5mm, 1mm, or at most 2mm, depending up on absorbance of the analyte. The water bands begin to dominate the spectra at path lengths over 0.5 to 1mm.

Polyol spectra may require longer path lengths, on the order or 4mm, 10mm, and 20mm. Because polyols generally contain very little water, the longer path lengths work well, and provide good detection of additives in the sample matrix.

Perform some initial analysis with cuvettes of various path lengths to determine optimal path length for your analysis.

Cuvette Type:

Open or capped cuvettes may be used with non-volatile compounds, where the constituents will remain in solution for the duration of analysis, and evaporation is not an issue.

Compounds with volatile ingredients should be analyzed in sealable cuvettes, to prevent loss of constituents to the atmosphere.

5.4 Flow-Through Fittings

Metrohm has provided flow-through bulkhead fittings on the door of the XDS Rapid Liquid Module for users who require analysis of flowing liquids.

These ports may be attached, using flexible tubing, to a suitable cuvette or cell which fits into the sample drawer. It is important to use tubing that is compatible with the analyte.
To access the outer fittings, gently lift the black cover and peel it back. The ports are exposed as shown. Save the cover as a light-block, in the event the ports are not used in later work. It should be re-installed to prevent light seepage through the fittings.

When using flowable cuvettes, the user should consider flow dynamics in the cell, along with safe connection of the ports to the cell inlet and outlet. Avoid spills and “blowouts” in the instrument.

5.5 Cuvette Lifting Handle

The Rapid Liquid Analyzer has a lifting handle to expedite removal of the cuvette from the sampling area. The photo at the right shows the handle in use.

Always use the lifting handle to remove cuvettes. DO NOT grasp the cap of the cuvette to lift it out. This may break the neck of the cuvette, and could expose the user to hazardous materials.

5.6 Cuvette Spacers

The Rapid Liquid Analyzer comes with a set of spacers for use with various size cuvettes. (The cuvette size is stamped on top of the spacer.) The spacers are designed to thread into a #10-32 tapped hole inside the sample drawer, to keep the spacer in the proper position.

Insert the spacer, then turn the knurled knob to thread it into the tapped hole in the sample drawer. Insert the cuvette carefully to avoid breakage. The drawer front is spring-loaded to permit easy insertion of the cuvette, and to provide secure positioning when in place.

5.7 Temperature Control Information

Setup of the Sample Temperature Control is discussed in section 7.3 of this manual. The controller is a heat-only control, and does not cool the sample in any way. Therefore, if samples are not presented to the instrument in a consistent manner, there may be some spectral effect from the elevated temperature. This spectral effect (due to temperature) may result in prediction errors.

The user should bear these items in mind:

- Always scan samples at a consistent temperature to avoid spectral variations due to temperature differences.
- If scanning at room temperature, be sure the samples have all equilibrated to that temperature.
• If scanning at an elevated temperature using the controller, be sure samples are not already above that temperature from prior processing, water baths, or for any other reason.

If in doubt about temperature consistency, check samples using a calibrated temperature device to determine that sample temperature is well-controlled.

5.8 Instructions for closing the sample drawer using Vision

When the XDS Rapid Liquid Analyzer will not be used for some time, the operator may wish to close the sample drawer. This prevents damage, and keeps airborne dust out of the sample drawer area.

The user must be connected to the instrument in Vision

Look at the bottom right corner of the screen. Note the temperature readout (27.0 in this example). Right-click on this icon. The menu shown will appear. Click on “Open/Close Drawer” to set the sample drawer in the correct position. Click once, and the drawer will move to the “other” position. When the sample drawer is in the desired position, disconnect from Vision.

5.9 Liquid Sampling Considerations

The XDS Rapid Liquid Analyzer is capable of detecting low levels of analytes in liquids. A brief overview of absorbance and pathlength considerations on a simple type of sample may be helpful.

A suitable pathlength must be selected for the sample, in expected concentration levels. This pathlength must be long enough that the analyte absorbs energy, and also must be short enough that the absorption is not masked by other factors.

First, determine the wavelength areas for analysis. Take spectra of the pure materials to see where the absorptions occur, without confusion from other components of the sample matrix.

Once the possible wavelength areas are determined, the sample should be run in cuvettes of different pathlengths to determine what pathlength gives the optimum analytical results. The example below illustrates some of the initial steps in this process.

In this example, we wish to measure 2-propanol in water, at concentrations up to 25% of 2-propanol. We take spectra of each material, to see where the absorbances occur.
The spectra show pure water and 99.5% pure 2-propanol, taken in 1mm pathlength cuvettes. Water absorbs strongly between 1350-1600nm and 1850-2150nm. 2-propanol has absorbances between 1650-1800nm, 2050-2150nm, and 2250-2500nm. 2-propanol is a weaker absorber than water; the spectral intensity is lower.

The 2050-2150nm absorbance in 2-propanol would probably be masked by the 1850-2150nm absorbance of water. 1mm pathlength is not a good choice.

Spectra of 25% 2-propanol in water, taken at 1mm and 2mm pathlengths, show the main absorber is water, with some absorbance due to 2-propanol. Vertical lines highlight the 2-propanol absorbances at 1690nm and 2310nm. Absorbance at 1690nm using a 1mm cuvette appears too low to be of much use.

Absorbance at 1690nm is about 0.55 absorbance units using a 2mm cuvette. If we work at that wavelength, a 4mm cuvette would provide a higher absorbance level.

The absorbance at 2310nm is about 2.6 absorbance units, using a 2mm cuvette, which might be fine on this instrument, if the sample gives linear absorbance. Some work should be done at each pathlength to determine which combination of wavelength area and pathlength gives the best analytical result.

Many factors may come into consideration, including mixing accuracy, lab error for samples analyzed by other methods, and operator differences. Contact your Metrohm NIRSystems Product Specialist for more information.
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 “direct connected” as explained in section 3.0 of this manual. Alternatively, the instrument and computer must both be plugged into a live RJ-45 communication jack, 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 will 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. For our purposes, we simply called this “project7”. Please use a more descriptive name.

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 “Acquire New Data”.

This screen sets up communication parameters for the instrument. The XDS instrument has a unique driver. Highlight this box and click on “Configure”.

This box allows the user to set the COM port and select the instrument IP (Internet Protocol) address.
Use the drop-down arrow of the IP Address box, and select the correct instrument. The instrument must show “Available” to be selected.

Click “OK” to exit the dialog box.

When preparing to run samples, the user may wish to enable “Blank Correction” as
described in section 7.1.3 of this manual. If so, this option must be set in Project Options. To do so, cancel out of Data Acquisition, and follow the steps on the next page. If you do not wish to use Blank Correction, continue with the Data Collection Method (DCM) selection.

Note that once a project and DCM have been set up without Blank Correction (and spectra have been stored using the DCM) it cannot be converted to Blank Correction. A new project and DCM must be created to allow the change in data collection.

Do not use Blank Correction during Diagnostics. It does not apply during Diagnostics.

To enable Blank Correction, cancel out of Data Acquisition, then click on Configure on the Vision menu bar.

Next, click on Options. Select Blank Correction as shown in this illustration.

When the Blank Correction box is checked in This Project’s Options, it sets the Data Collection Method (DCM) for use of Blank Correction with the Rapid Liquid Analyzer. This is explained in section 7.1.3.

Click “OK” to exit.

Select Acquire from the Vision Menu bar to proceed to the next step. Click on Connect.
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 Rapid Liquid system. The user should enter a logical name for the DCM.

The Equilibration Time may be set for the number of seconds required for samples to reach the desired temperature setting. Measure several typical samples to determine this time, then enter it into the DCM. Add several seconds to be sure temperature is reached. When this field is used, Vision delays by this amount of time before taking a sample scan. The default time is 0 seconds, as shown here.

Use 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 may check test parameters, Click the “Test Param” button, then select the wavelength area of interest. Specifications will be displayed.

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.

Upon successful connection, the sample drawer will quietly open. Do not manually force the sample drawer to either an open or closed position. It is under software control.

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.

Vision reads the sample module serial number and enters it in the blank field. Verify that this number is correct. It is important that the instrument be correctly identified, to prevent corruption of the database.

If correct, 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.

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.

Run performance test after wavelength linearization:

Forces user to run test sequentially. This is not necessary with XDS.

Reference Standardization:

Not used with the XDS Rapid Liquid Analyzer.

Use Auto-Linearization:

Maintains correct wavelength registration automatically, using internal wavelength materials to keep instrument in precise adjustment over time.

Blank Correction:

Used to create a correct transmission reference, when using cuvette spacers and other devices. Takes a scan of the sample chamber and the reference chamber, then applies a correction factor. This is explained in a later section. Recommended for Calibration Transfer.

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 Calibration Transfer.

Use Window Correction (XDS only)
This is only used with specific XDS process Instruments. Do not select this option.

When calibration or library transfer (between instruments) is anticipated, be sure that Blank Correction 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 Instrument Identification

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.

The monochromator Instrument Serial Number and the Sample Module Serial Number are read from stored information inside the instrument.

Click “OK” to accept the instrument identification.

7.1.2 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.

This test is run automatically as part of Instrument Calibration. The user must confirm the acceptance of the linearizations. Click “OK” to accept for each wavelength region.

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 standards are used to maintain the external wavelength registration by use of software adjustment for any external effects on the instrument.

Select Wavelength Linearization from the Diagnostics menu. The instrument will scan the reference.
The results screen shown above is typical. 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.

This is done twice, once for each direction of grating motion.

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

Click “OK” to proceed.

7.1.3 Blank Correction

The Rapid Liquid Analyzer has the option of “Blank Correction” to provide an optimum photometric match between instruments. This must be selected in Project Options. A software algorithm applies the correction to each spectrum automatically.

Blank correction has been shown to substantially minimize slight photometric differences between instruments, permitting simplified transfer of equations from one instrument to another.

Select “Create Blank Correction” from the menu. Vision gives on-screen, step-by-step instructions for the creation.
The menu selection for Blank Correction is available when the option is selection in This Project’s Options, and the Data Collection Method (DCM) has the Blank Correction box is checked. This is explained in section 7.1.2.

Upon selection of Blank Correction from the Diagnostics menu, Vision takes an instrument reference.

Next, this dialog box prompts the user to remove any cuvettes, and verify placement of the proper spacer.

When instructed, insert the correct spacer into the sample drawer and tighten the knurled knob fully.

**Be sure to use the right spacer for the cuvette size, or the Blank Correction will give the wrong spectral correction.**

Click “OK” when the spacer is in position. Vision will scan the sample area.

After the sample area is scanned, a dialog box prompts that Vision is ready to plot the Blank Correction. Click “OK” to proceed.

The spectrum at the right is a Blank Correction, and is a typical shape. The shape may vary slightly, depending upon the spacer used.

Vision applies the correction automatically to all subsequent spectra taken with this Data Collection Method (DCM), during this connection session. Upon disconnecting, the Blank Correction is erased.

**If the DCM is used for a different size cuvette, Blank Correction must be repeated with the new spacer.**

When finished, click on “Close Report” to proceed.

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**7.1.4 Instrument Calibration**

Instrument Calibration uses a traceable, stable, rare-earth glass standard of known wavelength response, as a method to establish wavelength scale response of the instrument. This standard is directly traceable to NIST SRM-2035. A spectrum of the standard is shown:
The black spectrum is SRM-2035. The gray spectrum is XC-1300, which is used to calibrate the wavelength scale of the XDS Rapid Liquid Analyzer.

Note that the XC-1300 standard used to calibrate the XDS Rapid Liquid Analyzer shares many of the same peak absorptions as SRM-2035. Both materials use some of the same rare earths, and the absorbances would naturally fall in the same places in the NIR spectrum.

The XC-1300 standard has additional peak absorbances, especially at the higher wavelengths. These additional wavelengths permit accurate calibration of the wavelength scale beyond that offered by SRM-2035.

A note about the XC-1300 Transmission Calibration Standard:

When first offered, the XDS Rapid Liquid Analyzer used a wavelength standard labeled WST3WCAL. It was later determined that better transferability between instruments could be achieved if each standard was calibrated on the Metrohm NIRSystems master transmission instrument. Since that time, each WST3Wxxx standard is serialized, with a matching calibration file. For example, the standard may now be labeled “WST3W068”. The calibration file is contained on a mini-CD that contains the file “TSS3W068” for that specific standard. Each serialized standard has a unique number.

The wavelength scale response of the instrument is established by instrument optical design. This is fine-tuned at the time of manufacture and testing. An initial wavelength assignment is made during Wavelength Linearization. In Instrument Calibration, the XC-1300 is used at the sample plane to make a final adjustment/verification of the wavelength response. By performing this function at the sample plane (where user samples will be scanned) the analytical response of the instrument can be assured.
To assure transferability of models between similar instrument models, one more step is taken as part of Instrument Calibration. The wavelength response of the instrument is corrected to conform to that of the Metrohm NIRSystems XDS master transmission instrument. The adjustment is quite small, normally less than several tenths of a nanometer. The adjustment is small enough to be negligible, but the effect on transferability (especially when using qualitative libraries) is noticeably improved.

At the same time, the “profile” of certain peaks is adjusted to conform to that of the master instrument. This adjusts for minor optical differences in the instrument “line shape” that cannot be controlled mechanically or optically, but which could affect transferability. These adjustments are applied on each subsequent scan of the instrument, yielding a correct sample spectrum. The method has been proven by comparing SRM-2035 sample scans on multiple instruments, with Instrument Calibration turned off, then on. The resulting scans are most consistent with Instrument Calibration turned on.

Instrument Calibration may be turned off by users who wish no such adjustment. The slight effect of Instrument Calibration on the spectrum is virtually beyond measurement using normal instrument techniques, yet has such a positive effect on transferability that Metrohm NIRSystems recommends it always be used with XDS instruments. It is set as a default with XDS instruments in Configure, Options.

The procedure for Instrument Calibration is as follows:

To start Instrument Calibration, select Instrument Calibration from the Diagnostics menu in Vision.

Insert the XC-1300 standard (labeled WST3W011 in this instance) into the sample drawer of the XDS Rapid Liquid Analyzer as shown.

Temperature of the sample drawer should be set for room temperature. (20 degrees C is typical.) Elevated temperatures will cause the wavelength positions to migrate slightly.
Vision takes an instrument reference, which takes about 20 seconds. This dialog box is displayed.

Next a sample scan is taken, and this box is displayed. This takes about 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. Continue until this step is completed.

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.

A dialog box confirms that this has taken place. Click “OK”.

Vision will perform these operations again, for the opposite direction of grating movement. Repeat the confirmations. Continue until the Wavelength Linearization steps are completed.
Following Wavelength Linearization, the user is asked to select a standard file. This is provided on the mini-CD found in the standards box, and may be read through the A: drive.

The file may be copied onto the computer in the C:\Vision directory, for convenience. (The file name will be similar to that shown, with a different serial number.)

Select this file and click on “Open”.

Vision prompts the user to insert the WST3W011 (or similar name) standard into the instrument.

Verify that the XC-1300 Wavelength Standard is in the proper position when prompted. 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. This is performed to assure good agreement from instrument to instrument, should multiple instruments be used for analysis of similar products.

When the test is finished, this box is displayed. Click “OK” to proceed.

This same XC-1300 standard will be used later during Wavelength Certification, to verify wavelength positions set during Instrument Calibration.

**It is important to note that two specific wavelength accuracy operations are performed during Instrument Calibration:**

1. Accuracy is measured against certain NIST-defined nominals, using the NIST uncertainty of +/- 1.0 nm.

2. Accuracy is also measured against tighter FOSS-defined wavelength parameters, which fall inside of NIST-defined parameters. The FOSS-defined parameters are typically +/- 0.05 nm for the XDS Rapid Liquid Analyzer.
These will be measured later in Wavelength Certification, as a test of how well the instrument is maintaining the settings established in Instrument Calibration.

7.1.5 IPV Setup (Instrument Performance Verification)

IPV Setup is provided as a method to record initial instrument response to calibrated photometric transmission 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. This operation uses the XC-1310 Standard Set.

When the standards are scanned during IPV Setup, a spectral 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 TSS30020, the IPV Setup file is named TSSV30020.

With the IPV Setup file stored, the user can later 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 Blank Correction 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 diskette into the A: drive, select that drive in the dialog box, and click on the TSS3xxx.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.

Vision displays the wavelength regions for test.

For XDS Rapid Liquid 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, retain the default setting of 1.

Click on “OK”.

Vision will begin to take an instrument reference scan. The red progress bar at the bottom of the screen indicates status.

Vision requests the T01 standard from the set.

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

Place the standard into the sample drawer, with the label facing upward.

Note the mini-CD that contains the “Standards File”. This file is used during IPV Setup. Once the IPV Setup file is created, Photometric Test will use a different file, which is stored in the Vision directory. Keep the mini-CD diskette with the standards. It is used in other tests.

Continue as requested by Vision, inserting the T02, T03, T04, and T05 standards when prompted. When finished, Vision reports “Test Successfully Completed”.
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 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.

Recent Vision software defaults to the “Trans Cal Std” (XC-1300) which gives more information than Polystyrene-Didymium type in the XC-1310 or XC-1320 set.

Note that the front (rare-earth) glass in the XC-1300 “Trans Cal Std” is reddish-pink in color, not green.

Select “Trans Cal Std.” as shown.

Click “OK” when ready.

Vision requests the Standard File for the wavelength standard. This file is located on the mini-CD in the XC-1300 standard set. It may be read using the CD/DVD drive, or may be copied into the C:\Vision directory as shown here.

The file name will be similar to that shown, using the format TSS3Wxxx.da, as shown here. The file contains the spectrum of the WST3Wxxx standard to be scanned.

Click “Open” to proceed.
When instructed, insert the WST3xxxx standard into the sample drawer.

The label should face the user when inserting the standard.

Place the standard into the drawer. The serial number should be readable from the front, as shown.

Click “OK” when ready.

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

This is a “typical” printout. It will be slightly different for each instrument.

Wavelength Certification (using the XC-1300 “Trans Cal Std.”) also tests certain FOSS-defined peaks in the wavelength standard. They are reported on the second page of Wavelength Certification, on a
page titled “Instrument Calibration Verification”. 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.

This is a “typical” printout. It will be slightly different for each instrument.

Note that Bandwidth is only computed on the XC-1300 WST3Wxxx “Trans Cal Std”, which is the same standard used in Instrument Calibration. (Shown on left.) The printout above is for WST3W011, and will be similar for other serialized XC-1300 wavelength standards.

Bandwidth is not calibrated or measured when using “Poly-Didym”. (Shown on right.) That standard also does not provide the tighter FOSS-defined peak readouts, as set in Instrument Calibration. It is included in XC-1310 and XC-1320 as an independent check from the XC-1300 standard, for those who desire a separate wavelength standard from that used in Instrument Calibration.

When Wavelength Certification 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.

If the instrument passes the NIST-type peaks (page 1 of the report), but fails on the FOSS-defined peaks, Vision will recommend that the user run Instrument Calibration to set the wavelengths properly.

Instrument Calibration must be checked under Project Options to get this message.

Verify that the instrument is fully warmed up, and run the test again. If this message is shown, re-run Instrument Calibration. This will set the wavelengths to the required values.

A note about Performance Test:

Performance Test measures peak positions of internally-mounted wavelength reference materials. 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, used in Wavelength Certification.

7.2.2 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.

As the test runs, a screen like that shown below is displayed. When finished, 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.

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 standard is disassembled, 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 TSSVxxxx.da file stored in the C:\Vision directory. This file was created during IPV Setup. Current photometric readings will be compared to that initial file.

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

Do not use the file on the standards diskette 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”.

Current versions of Vision include a newer version of this tolerance file, which may be named “IPVXDS02, 03, or higher. These files support additional XDS instruments, though that will not be visible to the user. The tolerances for the XDS Rapid Liquid Analyzer remain the same in all versions.

Vision displays the wavelength regions for test.

For Rapid Liquid 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, retain the default setting of 1.

Click on “OK”.

Vision will take an instrument reference scan, with the sample drawer in the open position. The red progress bar at the bottom of the screen indicates status.

Vision requests the T01 standard from the set, as shown.
Select the requested standard from the set. Labels on the top of each standard identify the number of the standard. Approximate transmission values are as follows:

- T01 0.25 AU (Absorbance Units)
- T02 0.50 AU
- T03 1.0 AU
- T04 2.0 AU
- T05 2.5 AU

Absorbance values vary across the spectrum, as well as from set to set. Always use the same set, with the labeled mini-CD.

Position the standard in the sample drawer with the label facing the user.

When the T01 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 T02, T03, T04, and T05 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”.

### 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 Rapid Liquid 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.

The view above shows a fairly typical XDS Rapid Liquid Analyzer. The gain program measures gain through the reference aperture, under control of Vision software. Gain readings are reported 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 is a sign that the lamp is burned out, or of some other type 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. It is no longer required by the United States Pharmacopeia (USP) General Information Chapter on Near-Infrared Spectroscopy.

Low Flux Test uses a nominal 10% transmission standard in the sample position. A noise test is run using this standard. Because the transmission value is less than the instrument standard, the test is considered a good method for testing instrument noise in the absorbance range of many common samples. The T03 standard has a nominal absorbance of 1.0 Absorbance Units.

The XDS instrument has an internal 10% neutral density (transmittance) screen, triggered by software, which can be used in place of an external 10% transmission 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. In most cases, the answer should be “No”. This defaults to an internal 10% screen which in permanently mounted in the instrument, and is positioned automatically for this test.

Click “Yes” to use an external 10% transmission standard.
XC-1310 Transmission Standard Set contains a 10% transmission standard (T033xxxx) which may be used for this test, if desired. The T03 transmission standard may be positioned in the sample drawer.

Vision closes the drawer to run the Low Flux Test when using an external sample (standard).

If the user clicks “No” to the external standard, then Vision will automatically trigger the 10% internal screen for this test. No user action is required.

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

**Low Flux Noise Test**

<table>
<thead>
<tr>
<th>Scan</th>
<th>EOC</th>
<th>F.R.P</th>
<th>Minimum</th>
<th>Maximum</th>
<th>Wavelength</th>
<th>Minimum</th>
<th>Maximum</th>
<th>Wavelength</th>
<th>Bias</th>
<th>RMS</th>
<th>Gain</th>
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<tr>
<td>1</td>
<td>0</td>
<td>0.82</td>
<td>0.33</td>
<td>2396</td>
<td>0.454</td>
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<td>0.055</td>
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<td>439</td>
<td>0.454</td>
<td>400</td>
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<td></td>
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<td>733</td>
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<td>382</td>
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<td>0.005</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td>0.133</td>
<td>0.073</td>
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<td></td>
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<tr>
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<td>-0.163</td>
<td>2478</td>
<td>0.312</td>
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<td>423</td>
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<td>1100</td>
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<td>0.009</td>
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<tr>
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<td>0.000</td>
<td>1100</td>
<td>0.000</td>
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<tr>
<td>seg 4</td>
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<td>-0.163</td>
<td>2478</td>
<td>0.312</td>
<td>2466</td>
<td>0.024</td>
<td>0.078</td>
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<td>418</td>
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<tr>
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<td>0.356</td>
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<td>0.052</td>
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<td>0.083</td>
<td></td>
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</tr>
</tbody>
</table>

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.

7.3 Temperature Control Setup

The XDS Rapid Liquid Analyzer uses a commercial temperature controller to maintain analysis temperature in the sample drawer. Temperature is read in degrees C.

A temperature sensing device is embedded in the drawer, very close to the cuvette, and this is used as a method to control the sample heaters.

This control is a “heat-only” type, and does not apply any cooling to the sample. It is important to set the temperature higher than the ambient temperature of any sample, to avoid errors due to temperature.

See section 5.6 for information and guidelines about use of the Temperature Control.

This photo shows the set point as 27.0 degrees C, and the actual sample temperature as 26.1 degrees C, approaching the set point. An equilibration delay may be set in Vision to permit the samples to reach present temperature before spectral acquisition. This is shown on the next page.

Follow this procedure to set sample temperature:

With the instrument powered on, press the INDEX button until the SP1 Indicator is lit. The green LED display will show “SP1”. The set point is shown in the Red LED display, and is adjusted using the arrow buttons.

Press the UP button to reach the desired set point for SP1. Temperature reads only in degrees Centigrade. Maximum allowable setting is 60 degrees C. (140 degrees F)

If the temperature setting is too high for SP1, use the down button to lower the set point.

When the set point is correct, press the ENTER button. The temperature will be controlled by the instrument. The display will reset to the normal display after about 30 seconds, after taking several sample temperature readings.

It is important to set the “Equilibration Time” in Vision for the amount of time needed to bring
samples up to the set point temperature. This is located in the Data Collection Method (DCM) as shown in the next illustration.

The Equilibration Time is measured in seconds. If the Equilibration Time is set for 30 seconds, the instrument will commence a sample scan 30 seconds after the operator clicks on the “Sample” icon.

The Equilibration Time should be long enough for every sample to reach the desired temperature. This varies by:

- Sample type (heat capacity)
- Cuvette volume
- Difference between ambient temperature and set point temperature

In practice it is best to check the equilibration time with a temperature probe in the cuvette, to determine the actual time required. When this is known, enter the value (with some reserve) into the DCM.

A value entered into the DCM cannot be changed after spectra have been stored using this DCM. This is to prevent changes in sample acquisition parameters, which could skew results for a given sample set.

If a different Equilibration Time is necessary, a new DCM must be created. This assures that the operator is aware of the change, and wishes it to be applied to all subsequent samples acquired under the new DCM.
8 Instrument Maintenance

Instrument maintenance is quite simple on the XDS Rapid Liquid 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.

![Diagram of instrument parts]

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.

Sampling areas should be kept clean to prevent loss of signal. In the Liquid Analyzer, avoid spills or debris inside the sampling drawer, which could soil the cuvettes when they are inserted into the holder.

Clean with a dampened, soft, non-linting tissue. Remove all liquid, surface deposits and dust.

Periodically verify that no equipment has been placed nearby that might produce vibration or impacts that could be transferred to the XDS Rapid Liquid 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 are 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 counterclockwise (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 black arrow on the lamp should point straight upward as shown. This is required for proper wavelength registration in Instrument Calibration.

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 Wavelength Linearization 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

9.1.1 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 Instrument Noise, Bandwidth, Wavelength Accuracy, Photometric Certification, 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.

9.1.2 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.

9.1.3 NIRStandards® for Instrument Performance Verification

Various regulatory bodies suggest (or require) regular testing of NIR instrumentation to verify continuing stability of photometric and wavelength response. These transmission standards operate with Vision Software. Vision 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 XDS Rapid Liquid Analyzers in the pharmaceutical industry.

The standards are calibrated on our Master Instrument for use with in Instrument Performance Verification.

9.1.4 Instrument Performance Certification

The United Code of Federal Regulations, Title 21, requires user 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.

### 9.1.5 Metrohm Master Instrument Program

In support of our worldwide base of instruments, Metrohm maintains a Near-Infrared Master Transmission 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.

Vision Validation Document Package

This Compact Disc (CD) contains full documentation of the Vision Spectral Analysis Software design process, as stated by the PhRMA. The Part Number is AP-0007, and is available for purchase through your Metrohm Distributor.

At 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.

9.2.3 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 Rapid Liquid 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 next 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 Rapid Liquid 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 Department (or your local authorized distributor) if the problem is not solved.

<table>
<thead>
<tr>
<th>Observed Problem</th>
<th>Recommendations</th>
</tr>
</thead>
</table>
| 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. |
<table>
<thead>
<tr>
<th>2</th>
<th>No communication between Vision and the instrument.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1. Verify that the RJ-45 cable is plugged in at both the instrument and at the network wall jack.</td>
</tr>
<tr>
<td></td>
<td>2. Verify that the RJ-45 cable is plugged in at both the computer and at the network wall jack.</td>
</tr>
<tr>
<td></td>
<td>(NOTE: Direct connection is explained in section 3.0 for non-network users. This requires a special cable.</td>
</tr>
<tr>
<td></td>
<td>3. Verify that the instrument is powered on. (See previous Observed Problem.)</td>
</tr>
<tr>
<td></td>
<td>4. Verify that the network wall jack is active, and has a connection point within the internal network.</td>
</tr>
<tr>
<td></td>
<td>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.</td>
</tr>
<tr>
<td></td>
<td>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.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>3</th>
<th>Lamp does not come on when instrument is connected.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>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.</td>
</tr>
<tr>
<td></td>
<td>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.</td>
</tr>
<tr>
<td></td>
<td>3. Instrument thermal shutdown may have occurred due to high internal operating temperatures. Determine cause of high temperature and correct before subsequent operation.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>4</th>
<th>Instrument fails Wavelength Linearization</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1. Verify that the sample drawer is not jammed or impeded in any way. It should be free to open and close under software control.</td>
</tr>
<tr>
<td></td>
<td>2. Instrumental problems – contact Metrohm service or your authorized distributor.</td>
</tr>
</tbody>
</table>
|   | Instrument Fails Performance Test. | 1. Verify that the sample drawer is not jammed or impeded in any way. It should be free to open and close under software control.  
   |   | 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. A weak lamp will exhibit higher noise near the end of its life. This may also show up as one or two peaks in Performance Test not passing. If warm-up does not help, consider a lamp change. A new lamp will often solve a variety of subtle problems. The Performance Test is the easiest way to track lamp condition – as the lamp begins to fail, noise goes upward. |
|   | Instrument fails Instrument Calibration. | 1. Verify that the sample drawer is not jammed or impeded in any way. It should be free to open and close under software control.  
   |   | 2. Verify that the Wavelength Standard is properly positioned in the sample drawer when prompted by software. |
|   | Instrument Gain is excessively high in Gain Test. | 1. NIR Gain should always be less than 10 on the reference. Higher gain indicates lamp is out, or instrument is in need of service.  
   |   | 2. Verify that lamp is lit. (see above) |
### 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 in 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.

### Instrument cooling fans not operating.

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.

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** 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.
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