

Brief Operating Instructions

Raman Rxn4





Table of Contents






1	About this document.....	4
1.1	Warnings	4
1.2	Symbols	4
1.3	U.S. export compliance.....	4
2	Basic safety instructions	5
2.1	Requirements for the personnel	5
2.2	Intended use	5
2.3	Workplace safety.....	5
2.4	Operational safety	6
2.5	Product safety	6
2.6	IT security	6
3	Product description	7
3.1	The Raman Rxn4 analyzer	7
3.2	Product design	9
4	Incoming product acceptance and identification	11
4.1	Incoming acceptance.....	11
4.2	Scope of delivery.....	12
5	Electrical connection.....	13
5.1	Port connections.....	13
5.2	Power and grounding.....	14
6	Commissioning.....	15
6.1	Electrical interconnect block diagrams.....	15
6.2	Hazardous area installation drawing.....	18
6.3	Interior of the Raman Rxn4.....	19
6.4	Air filter	20
7	Operation.....	21
7.1	Raman RunTime embedded software.....	21
7.2	Initial Raman RunTime setup.....	21
7.3	Calibration and verification.....	23
8	Diagnostics and troubleshooting.....	25
8.1	Warnings and errors	25
8.2	Contact information.....	25

1 About this document

1.1 Warnings

Structure of Information	Meaning
<div> WARNING</div> <div>Causes (/consequences) Consequences of non-compliance (if applicable) ► Corrective action</div>	This symbol alerts you to a dangerous situation. Failure to avoid the dangerous situation can result in a fatal or serious injury.
<div> CAUTION</div> <div>Causes (/consequences) Consequences of non-compliance (if applicable) ► Corrective action</div>	This symbol alerts you to a dangerous situation. Failure to avoid this situation can result in minor or more serious injuries.
<div>NOTICE</div> <div>Cause/situation Consequences of non-compliance (if applicable) ► Action/note</div>	This symbol alerts you to situations which may result in damage to property.

1.2 Symbols

Symbol	Description
	The Laser Radiation symbol is used to alert the user to the danger of exposure to hazardous visible laser radiation when using the system.
	The High Voltage symbol that alerts people to the presence of electric potential large enough to cause injury or damage. In certain industries, high voltage refers to voltage above a certain threshold. Equipment and conductors that carry high voltage warrant special safety requirements and procedures.
	The CSA Certification Mark indicates that the product was tested against and met the applicable North American standards requirements.
	The WEEE symbol indicates that the product should not be discarded as unsorted waste but must be sent to separate collection facilities for recovery and recycling.
	The CE Marking indicates conformity with health, safety, and environmental protection standards for products sold within the European economic area (EEA).

1.3 U.S. export compliance

The policy of Endress+Hauser is strict compliance with U.S. export control laws as detailed on the website of the [Bureau of Industry and Security](#) at the U.S. Department of Commerce.

2 Basic safety instructions

Read this section carefully to avoid danger to individuals or the facility. Additional laser safety information and hazardous area certification and safety instructions are contained in the *Raman Rxn4 Safety Instructions (XA02745C)*.

2.1 Requirements for the personnel

- Installation, commissioning, operation, and maintenance of the measuring system may be carried out only by specially trained technical personnel.
- Technical personnel must be authorized by the plant operator to carry out the specified activities.
- Electrical connections may be performed only by an electrical technician.
- Technical personnel must have read and understood these Brief Operating Instructions and must follow the instructions contained herein.
- Faults at the measuring point may only be rectified by properly authorized and trained personnel. Repairs not described in this document must be carried out only directly at the manufacturer's site or by the service organization.

2.2 Intended use

The Raman Rxn4 analyzer is recommended for use in the following applications:

- **Chemical:** Reaction monitoring, blending, feed, and final product monitoring
- **Polymer:** Polymerization reaction monitoring, polymer blending
- **Pharmaceutical:** Active pharmaceutical ingredient (API) reaction monitoring, crystallization, polymorph, drug substance production unit operation
- **Oil and Gas:** Any hydrocarbon analysis

Use of the device for any purpose other than that described poses a threat to the safety of people and of the entire measuring system and invalidates any warranty.

2.3 Workplace safety

- Do not use the Raman Rxn4 for anything other than its intended use.
- Do not drape the power cord over counters, on hot surfaces, or in areas where damage to the integrity of the power cord may occur.
- Do not open the Raman Rxn4 unless you have been specifically trained on service and maintenance of the unit.
- Do not look directly into the laser beam.
- Do not allow emitted laser light to reflect off mirrored or shiny surfaces in an uncontrolled way.
- Minimize the presence of shiny surfaces in the working area and always use a laser beam block to prevent uncontrolled transmission of the laser light.
- Do not leave unused probes uncapped or unblocked while they are still attached to the analyzer.

2.4 Operational safety

Before commissioning the entire measuring point:

1. Verify that all connections are correct.
2. Ensure that electrical cables and optical fiber connections are not damaged.
3. Do not operate damaged products. Protect them against unintentional operation.
4. Label damaged products as defective.

During operation:

1. If faults cannot be rectified, products must be taken out of service and protected against unintentional operation.
2. Keep the door closed and properly sealed on the optional enclosure when not carrying out service and maintenance work.



Activities while the analyzer is in operation introduce risk of exposure to hazardous materials.

- ▶ Follow standard procedures for limiting exposure to hazardous chemical or biological materials.
- ▶ Follow workplace policies on personal protective equipment including wearing protective clothing, goggles, and gloves and limiting physical access to analyzer location.
- ▶ Clean any spills following the appropriate site policies and cleaning procedures.



There is risk of injury from the analyzer door stop mechanism.

- ▶ If the optional analyzer enclosure needs to be open, always open the enclosure door fully to ensure the doorstop engages properly.

2.5 Product safety

The product is designed to meet local safety requirements for the intended application, and has been tested accordingly, leaving the factory in a condition in which it is safe to operate. All applicable regulations and international standards have been observed. Devices connected to the analyzer must also comply with the applicable safety standards and users should follow the probe-specific product safety instructions.

2.6 IT security

Our warranty is valid only if the device is installed and used as described in the Operating Instructions. The device is equipped with security mechanisms to protect it against any inadvertent changes to the settings.

IT security measures, which provide additional protection for the device and associated data transfer, must be implemented by the operators themselves in line with their security standards.

3 Product description

3.1 The Raman Rxn4 analyzer

The Raman Rxn4 analyzer, powered by Kaiser Raman technology, is a for-purpose embedded system with built-in Raman RunTime control software. Raman spectroscopy provides the chemical specificity of mid-infrared (IR) spectroscopy and the sampling simplicity of near-IR spectroscopy. By operating in the visible or near-IR spectral region, Raman spectroscopy allows vibrational spectra to be easily collected *in situ*, using fiber-coupled probes, without sample purging, and without the use of specialty sampling devices.

There are three possible configurations of the Raman Rxn4 analyzer: single-channel, four-channel, and hybrid. All Raman Rxn4 analyzers employ a unique self-monitoring system to ensure the validity of each analysis. The analyzer is capable of two-point self-calibration in extreme environments and utilizes self-diagnostics and spectral correction methods when system calibration is unnecessary. The analyzer's precision is essential for robust chemometric analyses and calibration transfer between instruments. The Raman Rxn4 suite of analyzers allows for remote fiber-optic connections to probe sampling points for installation flexibility. And all configurations of the Raman Rxn4 analyzer are designed for use with the Endress+Hauser line of Raman fiber-optic probes and optics.

3.1.1 The Raman Rxn4 single-channel and four-channel configurations

The Raman Rxn4 single-channel configuration offers one fiber-optic probe connection for the accurate measurement of a single point in a continuous or batch process. The Raman Rxn4 four-channel configuration provides four fiber-optic sample probe connectors to sequentially measure up to four different *in situ* sample points in continuous or batch processes in the life sciences, chemical, oil and gas, and food and beverage industries. Raman Rxn4 single-channel or four-channel configurations are available with a 532 nm, 785 nm, or 993 nm excitation wavelength laser.

With the Raman RunTime software running on the embedded controller, the Raman Rxn4 single-channel and four-channel configurations meet the needs of both good laboratory practice (GLP) and good manufacturing practice (GMP) governed areas within the pharmaceutical industry for process analytical technology (PAT) and quality by design (QbD) applications.

3.1.2 The Raman Rxn4 hybrid configuration

The Raman Rxn4 hybrid configuration is unique because it contains connectors for an Rxn-20 large volumetric probe and a second, alternate (ALT) backscattered probe. The Raman Rxn4 hybrid configuration is only available with a 785 nm excitation wavelength laser.

The two different probe types enable a variety of applications for solids, liquids, and turbid media. A backscattered immersion probe is the preferred approach for measuring liquids because of its short focus, optical window, and bubble-shedding design. The Rxn-20 probe is optimized for large volumetric measurements, enabling focus-free, non-contact

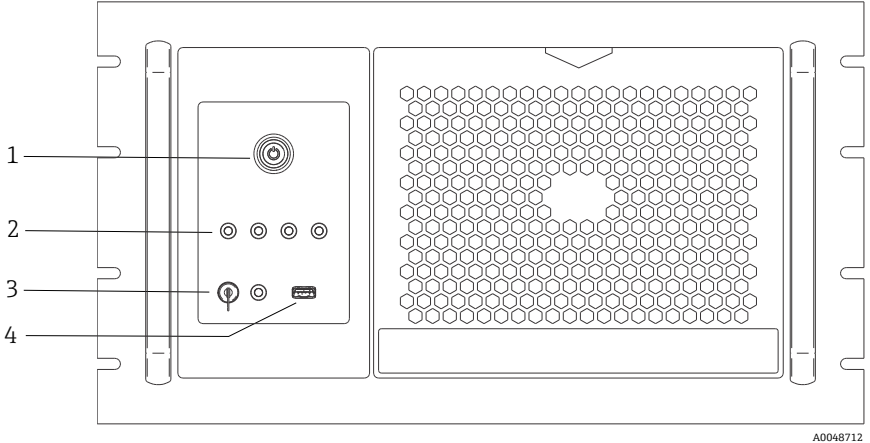
representative measurements of solids or turbid media. The hybrid configuration provides maximal sampling flexibility for *in situ* analysis of continuous or batch processes.

With Raman RunTime analyzer control software running on the embedded controller inside the analyzer, the Raman Rxn4 hybrid configuration meets the needs of both good laboratory practice (GLP) and good manufacturing practice (GMP) governed areas within the pharmaceutical industry for process analytical technology (PAT) and quality by design (QbD) applications.

3.2 Product design

3.2.1 Front panel

On the front panel of the instrument are the standard user interfaces. These include the main **ON/OFF** power switch, the laser **ON/OFF** key switch, Light emitting diode (LED) indicators, and a universal serial bus (USB) 3.0 port.



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Figure 1. Front panel of a Raman Rxn4 four-channel analyzer

#	Name	Description
1	Main power switch	The main power switch turns the instrument on and off, which includes the laser regardless of the position of the laser key switch. The Power push button incorporates a Blue LED in the shape of a power symbol, which indicates the system power status (all components are powered when lit). The Power push button will communicate error conditions using blink codes when embedded software is not able to communicate them. To turn the instrument on, press and release the Power button once. To turn a responsive instrument off, shut down using Raman RunTime. If the instrument is unresponsive, it may be powered down using a long 10 second press and hold of the Power button.
2	Probe connection status indicators	The bank of Yellow LED indicators above the laser key and USB 3.0 port indicates the physical connection status of each probe. The LED is lit when the corresponding probe is properly connected. While the Raman Rxn4 four-channel configuration front panel has four LED indicators, the front panel of the Raman Rxn4 hybrid configuration has only two LED indicators, and the front panel of the Raman Rxn4 single-channel configuration has only one LED indicator.

#	Name	Description
3	Laser key switch	The laser key switch turns the laser on and off. The Red LED indicator adjacent to the laser key switch indicates the laser power status. To activate the laser, turn the laser key switch to the ON position. The Red LED indicator should remain lit whenever the laser is ON.
4	USB 3.0 port	The USB 3.0 port is intended to obtain diagnostic exports from the instrument using a USB flash drive.

3.2.2 Rear panel

On the rear panel of the instrument are the standard ports. These include touchscreen, USB, Ethernet, serial, and video ports.

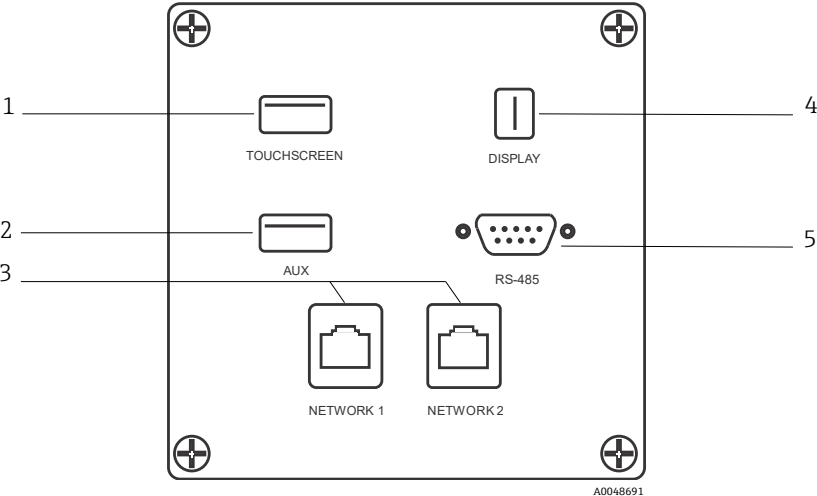


Figure 2. Rear external circuit input/output panel of a Raman Rxn embedded analyzer

#	Name	Description
1	Touchscreen USB port	USB 2.0 port used to connect to the touchscreen.
2	USB port (auxiliary)	USB 2.0 backup port. Reserved for future use.
3	Ethernet port (2)	Ethernet ports for the network connection.
4	Touchscreen video port	Touchscreen video port for connection to local touchscreen display (if needed).
5	RS-485 serial port	RS-485 serial port, half-duplex. Provides automation data via Modbus remote terminal unit (RTU). Port settings configurable in Raman RunTime.

4 Incoming product acceptance and identification

4.1 Incoming acceptance

1. Verify that the packaging is undamaged. Notify the supplier of any damage to the packaging. Keep the damaged packaging until the issue has been resolved.
2. Verify that the contents are undamaged. Notify the supplier of any damage to the delivery contents. Keep the damaged goods until the issue has been resolved.
3. Check that the delivery is complete and nothing is missing. Compare the shipping documents with your order.
4. Pack the product for storage and transportation in such a way that it is protected against impact and moisture. The original packaging offers the best protection. Make sure to comply with the permitted ambient conditions.

If you have any questions, refer to our website (<https://endress.com/contact>) for the list of local sales channels in your area.

NOTICE

Incorrect transportation can damage the analyzer.

- Always use a lifting truck or a forklift to transport the analyzer.

4.1.1 Nameplate

The nameplate located on the rear of the analyzer provides the following information about your device:

- Manufacturer contact information
- Laser radiation notice
- Electric shock notice
- Model number
- Serial number
- Wavelength
- Maximum power
- Build month
- Build year
- Patent information
- Certification information

Compare the information on the nameplate with the order.

4.1.2 Identifying the product

The order code and serial number of your product can be found in the following locations:

- On the nameplate
- In the delivery papers

4.1.3 Manufacturer address

Endress+Hauser
371 Parkland Plaza
Ann Arbor, MI 48103 USA

4.2 Scope of delivery

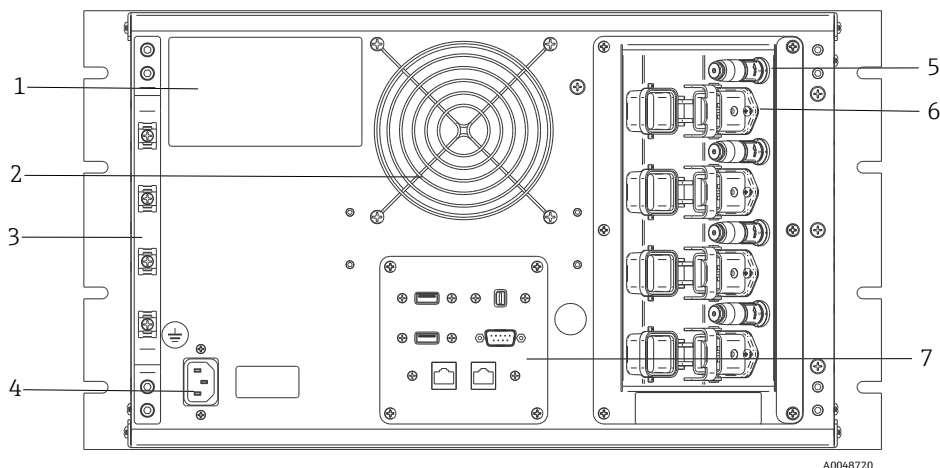
The scope of delivery comprises:

- Raman Rxn4 analyzer in the configuration ordered
- *Raman Rxn4 Operating Instructions*
- *Raman RunTime Operating Instructions*
- Raman Rxn4 Certificate of Product Performance
- Local declarations of conformity, if applicable
- Certificates for hazardous zone use, if applicable
- Raman Rxn4 optional accessories, if applicable

If you have any questions regarding the items delivered, or if anything appears to be missing, refer to our website (<https://endress.com/contact>) for the list of local sales channels in your area.

5 Electrical connection

5.1 Port connections



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Figure 3. Rear panel of Raman Rxn4

#	Name	Description
1	CDRH product label	Product information about the Raman Rxn4 analyzer
2	Air exhaust	Air exhaust fan and outlet
3	Strain relief	EO fiber cable strain relief mounting location
4	AC inlet 100 to 240 V 50/60 Hz	Power socket outlet that provides AC power to the base unit. Ground pin on this connector serves as the protective conductor terminal.
5	Remote interlock connectors	Safety feature. To interrupt the laser, remove the black plug.
6	EO fiber connector	Provides fiber-optic laser radiation output, fiber-optic Raman collection and electrical laser interlock loop for each instrument channel. The electrical laser interlock loop is intrinsically safe and is governed by Endress+Hauser drawing 4002396. Match 3 prongs on probe to 3 plugs on EO. Pull latch down to secure probe in place. Laser radiation may NOT exit a channel whose EO fiber connector is removed because removing the EO connector also interrupts the laser interlock loop for that channel.
7	Analyzer ports	Touchscreen USB port, USB port, Ethernet ports, RS-485 serial port, and touchscreen video port

5.2 Power and grounding

The Raman Rxn4 has a standard IEC-320 C-14 inlet for power at the rear of the instrument. Any power cord with an IEC-320 C-13 plug will connect to the base unit. The Raman Rxn4 accepts AC power from 100 to 240 V and 50/60 Hz. For U.S. applications, a power cord is supplied. For non-U.S. applications, the user must supply a power cable that meets local/national standards.

Also included on the rear of the instrument is a functional earth terminal for additional grounding if required. Primary grounding occurs through the IEC power inlet plug ground terminal which should be connected to the building grounding system.

Do not position the Raman Rxn4 in a way that makes it difficult to remove the mains cord. Only use adequately rated power cables with the Raman Rxn4 system.

6 Commissioning

6.1 Electrical interconnect block diagrams

6.1.1 Raman Rxn4 single-channel configuration

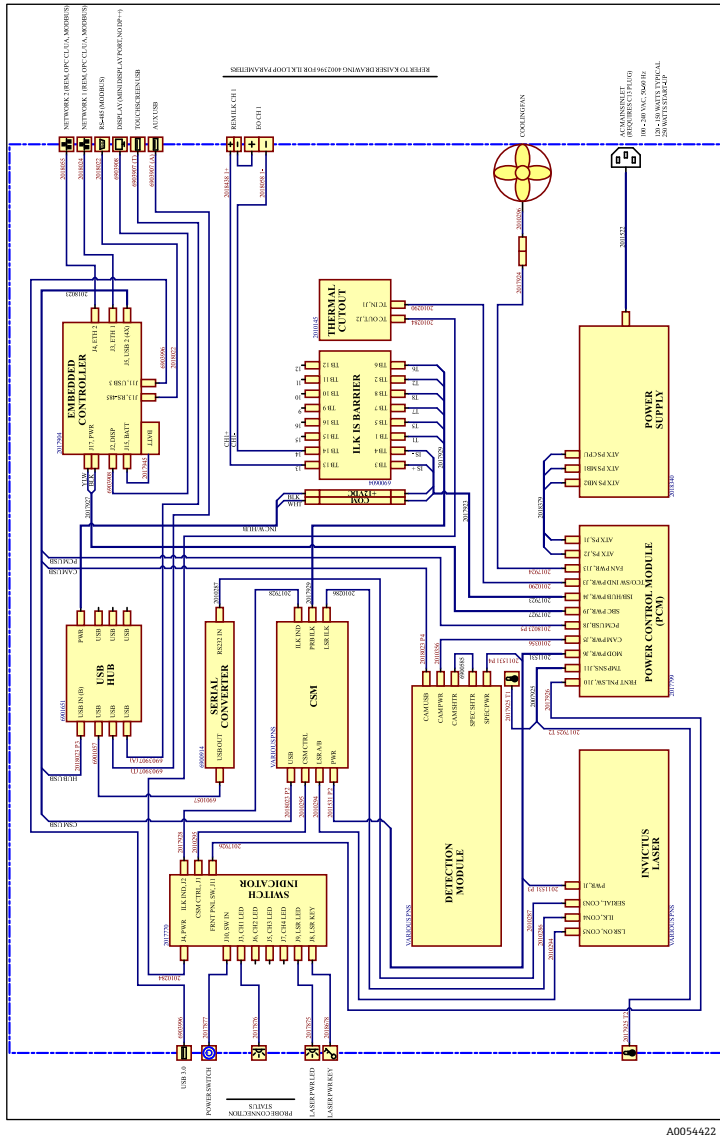


Figure 4. Raman Rxn4 single-channel configuration

6.1.2 Raman Rxn4 four-channel configuration

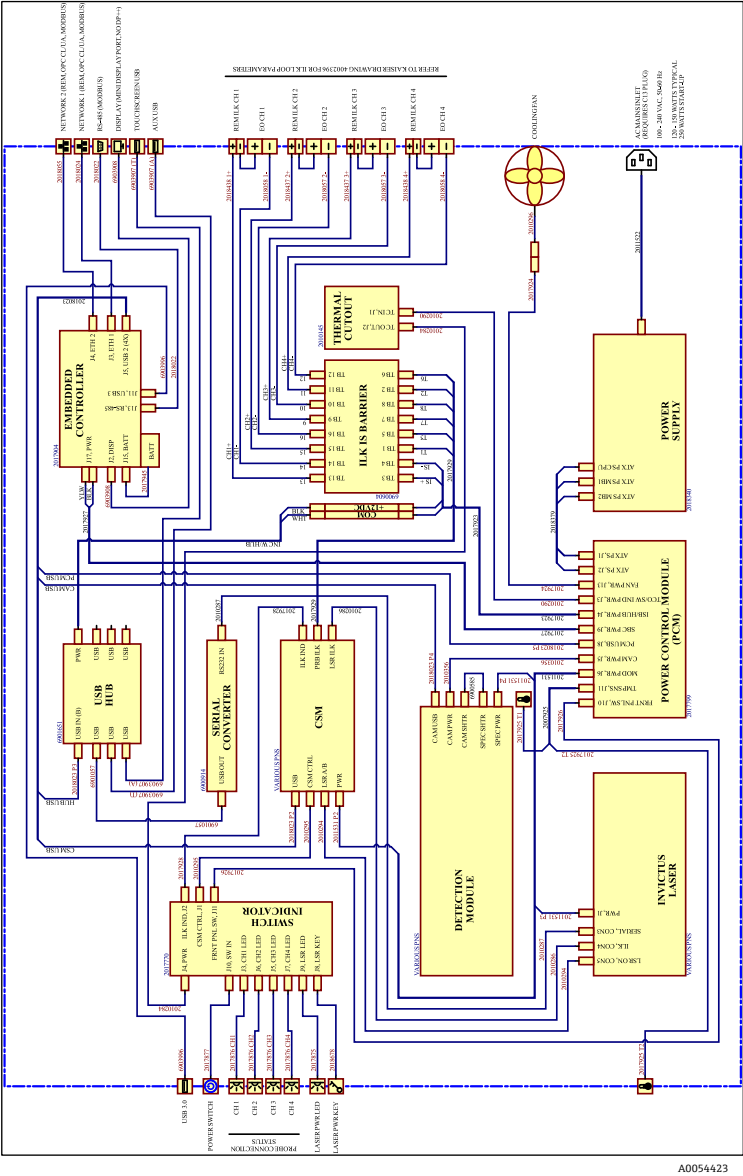
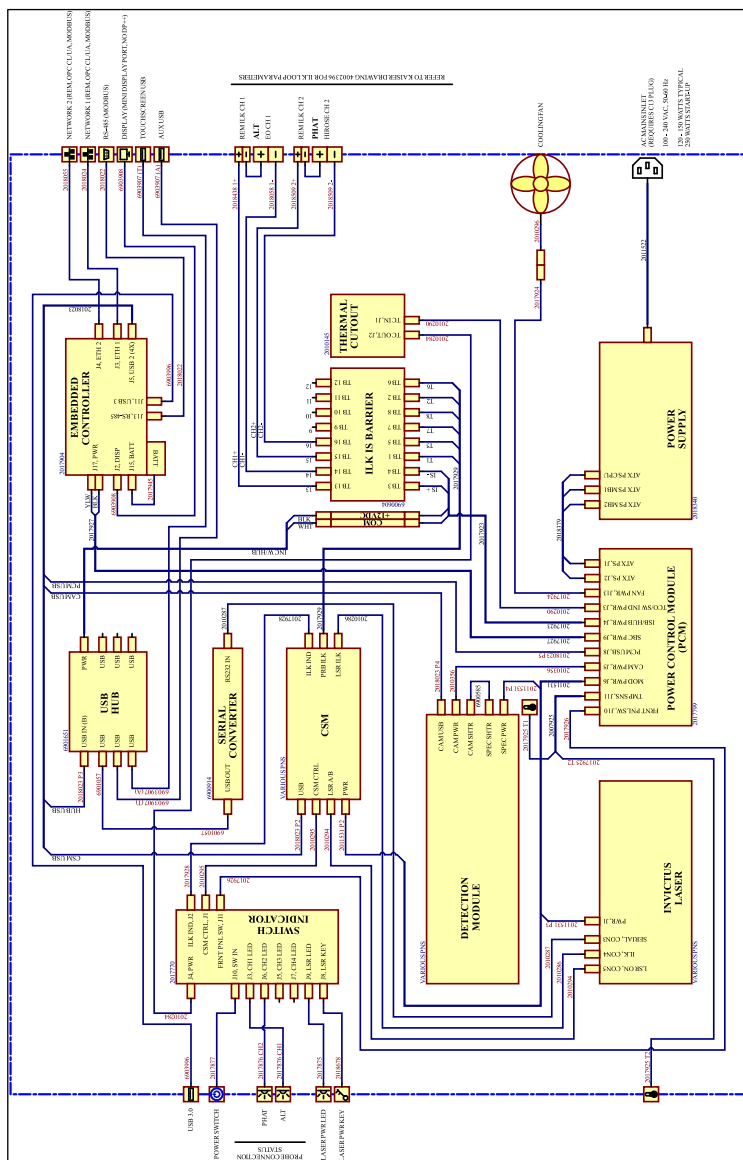


Figure 5. Raman Rxn4 four-channel configuration

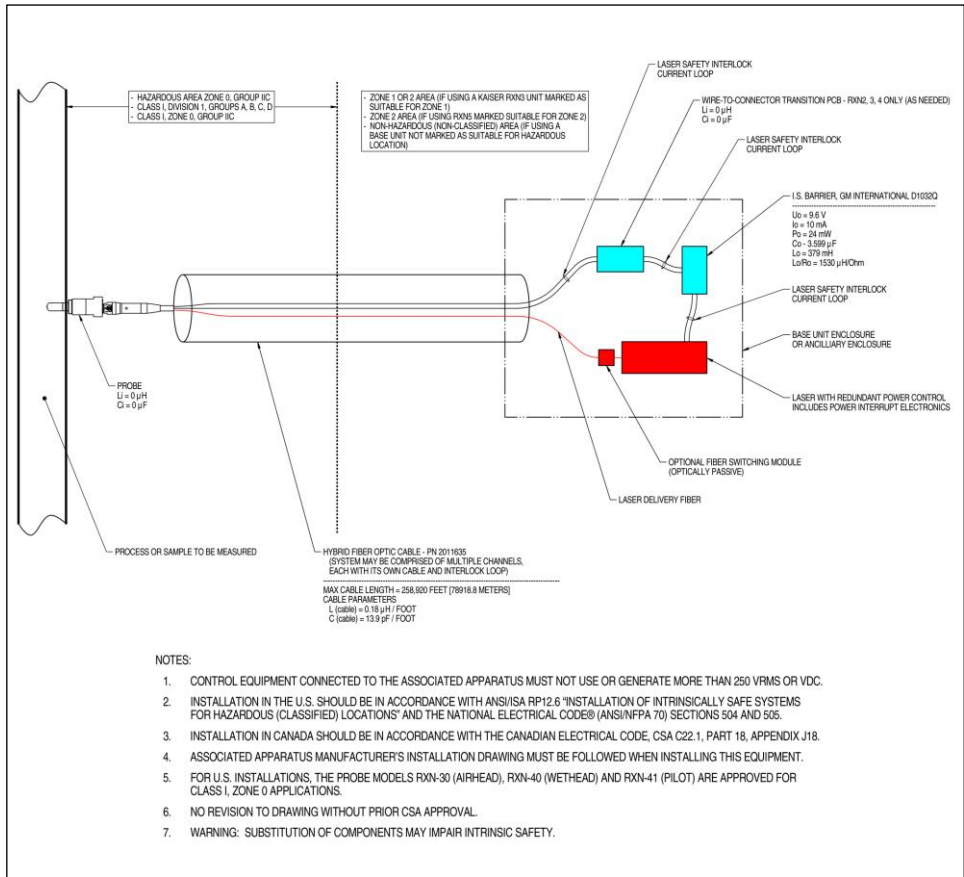
6.1.3 Raman Rxn4 hybrid configuration



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Figure 6. Raman Rxn4 hybrid configuration

6.2 Hazardous area installation drawing

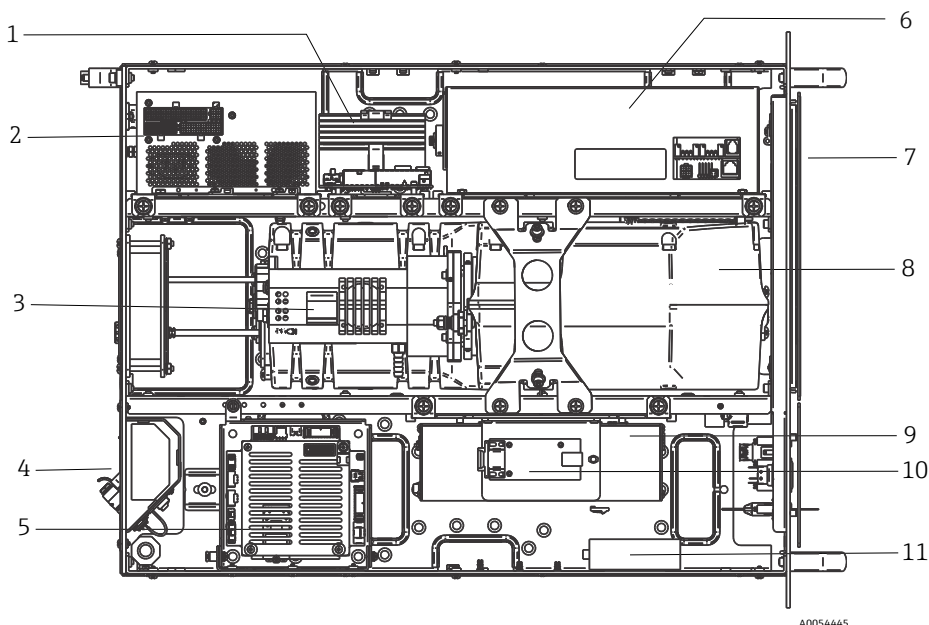


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Figure 7. Hazardous area installation drawing (4002396 X6)

6.3 Interior of the Raman Rxn4

The interior of the Raman Rxn4 with the cover removed is shown below. The internal components are common among all configurations.



A0054445

Figure 8. The interior of the Raman Rxn4 analyzer

#	Description
1	Power control module
2	Power supply
3	Internal temperature sensor
4	Excitation and collection fiber optics
5	Embedded controller
6	Laser module
7	Air inlet with incorporated ambient temperature sensor
8	Spectrograph module
9	CSM module
10	Serial converter
11	USB hub

6.4 Air filter

The Raman Rxn4 incorporates a tacked polyester spun air filter element to reduce dust intake into the base unit. The air filter is accessed by a magnetically secured access panel on the front of the instrument. The air filter should be cleaned with compressed air once every month or if the embedded software is reporting an internal over-temperature error (if ambient temperature is within specification). In extremely dusty conditions, the air filter should be cleaned more often. The air filter has a blue tacky side which should be oriented toward the outside of the base unit.

If a replacement air filter (p/n 70199233) is needed, refer to our website (<https://endress.com/contact>) for the list of local sales channels in your area.

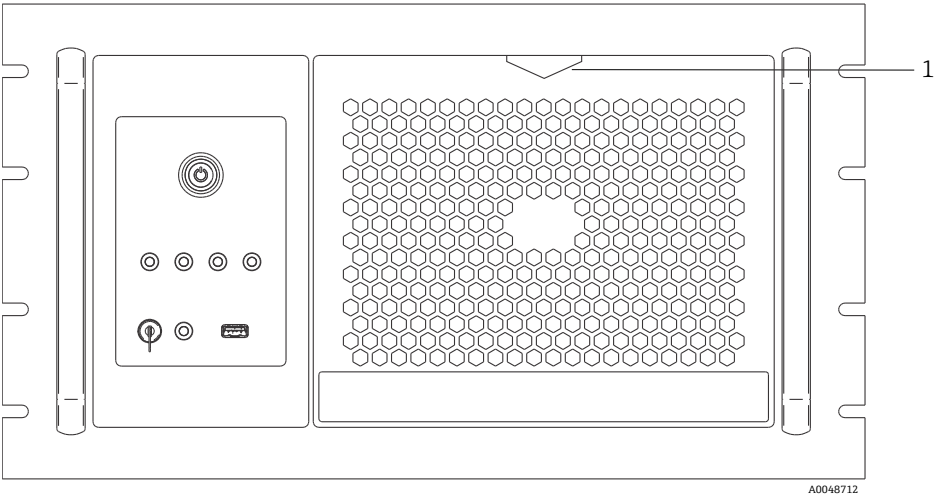


Figure 9. Pull (1) for access to air filter

7 Operation



WARNING

- ▶ Whenever the Raman Rxn4 analyzer main power switch and laser key are in their **ON** position, probes should be shuttered or covered, or remain immersed in the sample to be measured.

7.1 Raman RunTime embedded software

Raman RunTime is the embedded control software installed on all the Raman Rxn4 analyzers. It is intended for easy integration with standard multivariate analysis and automation platforms to enable a real-time, *in situ* process monitoring and control solution. Raman RunTime presents an OPC and Modbus interface which provides clients with analyzer data as well as analyzer control functions. Refer to the *Raman RunTime Operating Instructions (BA02180C)* for complete instructions on configuring and using the Raman Rxn4 with Raman RunTime.

7.2 Initial Raman RunTime setup

To perform initial Raman RunTime software setup, follow the instructions below.

1. Customize the analyzer name. The default name is “Raman Analyzer”:
 - From the Raman RunTime dashboard, navigate to **Options > System > General**.
 - Click the **Instrument Name** field.
 - Enter a custom name, for example, Raman Rxn4-785 sn0012345, then click **Apply**. The analyzer name is how the system is identified in diagnostic exports and within calibration reports.
2. (Optional) Calibrate the touch screen:
 - From the dashboard, navigate to **Options > System > General > Calibrate Touch Screen**.
 - Follow the on-screen prompts. To achieve better calibration, use the edge of your fingernail when following on screen prompts and touching the requested touch points.

3. Customize the identity for communication protocols, and customize network settings:
 - Navigate to **Options > System > Network**.
 - Click the **Hostname** field.
 - Enter a custom name and click **Apply**. This is a critical step because the hostname is how the Raman Rxn system is identified through communication protocols.
If using DHCP, the IP address is obtained automatically.
 - (Optional) Enter the static IP information, as applicable, then click **Apply**.
4. Set the date and time:
 - From the dashboard, navigate to **Options > System > Date & Time**.
 - Specify the time, date, and time zone, or
 - Enable **Time Synchronization**. Provide a time server address on the local network.
 - Click **Apply**.
 - ▶ If setting the date and time manually, ensure the time zone is set up correctly before proceeding to other adjustments.
 - ▶ This is another critical step because spectral acquisition and resulting files and communication protocols are managed by the system's date/time.
5. Specify names for each probe/quadrant such as Probe 1, Probe 2:
 - From the dashboard, click the title bar of the probe you wish to name. The stream or probe detail view displays.
 - Select the **Settings Tab** and click **Name**.
 - Enter the name of the probe and click **Apply**.
 - Let the system stabilize for at least two hours before proceeding to calibration.
6. Refer to the *Raman RunTime Operating Instructions (BA02180C)* for initial calibration and verification instructions.

7.3 Calibration and verification

Reliable, transferable calibration is important for comparing data acquired at various times or with different analyzers. Different instruments analyzing the same sample can generate nearly identical spectra if they are properly calibrated. The Raman RunTime software package includes an automatic calibration wizard that guides you through a procedure to automatically calibrate the wavelength and intensity axes and the wavelength of the laser.

After the initial calibration during installation, the Calibrate Periodically function is usually sufficient to maintain the wavelength and laser calibration of the Raman Rxn4.

A summary of the recommended calibration and verification sequence is as follows:

1. Internal calibration
2. Probe calibration
3. Probe verification

7.3.1 Internal calibration

Raman Rxn4 analyzers have internal calibration standards for spectrograph and laser wavelength. The internal calibration options are:

- **Automatic.** If the instrument is already calibrated, this setting compares the current analyzer response to calibration specifications and applies an algorithmic correction if it is minorly out of specification. This setting also recalibrates if the spectrograph wavelength, laser wavelength, or both are out of specification. If the analyzer is uncalibrated, this performs an alignment calibration, followed by a full wavelength calibration and full laser wavelength calibration.
- **Recalibrate X Axis.** Forces full wavelength and laser calibrations without first checking whether the analyzer is within spec.
- **Recalibrate All.** This setting causes the alignment calibration to be repeated prior to performing full spectrograph wavelength and laser wavelength calibrations. Note that when **Recalibrate All** completes, the intensity calibrations and verifications of all probes are invalidated.

Refer to the Calibration and verification section of the *Raman RunTime Operating Instructions (BA02180C)* for steps to perform or set periodic internal calibrations.

7.3.2 Probe calibration

The sensitivity of the Raman Rxn4 varies with wavelength due to variations in the throughput of the optics and the quantum efficiency of the CCD. The probe calibration function in Raman RunTime can be used to remove the effects of this variation from measured spectra.

Probe calibration can be performed using a probe-specific calibration kit or the HCA Raman calibration accessory. Refer to the applicable probe or optics manual to determine the appropriate calibration accessory. Refer to the calibration accessory manual for details on how to calibrate the specific analyzer/probe combination. Each channel must be probe calibrated separately.

Probe calibration can be performed during active experiments, for example when a probe needs to be set up while another probe is active. When a probe calibration is triggered, any acquisitions currently in progress are aborted automatically, and the calibration proceeds. Upon completion of the calibration, active probes automatically resume normal operation.

7.3.3 Probe verification

The probe verification wizard may be used to verify that the Raman Rxn4 is performing within specifications. Probe verification acquires a Raman spectrum of a standard Raman sample, usually 70 % IPA or cyclohexane, and analyzes the resultant spectrum for peak positions, peak area ratios and Raman signal strength. The peak position verification confirms that the spectrograph and laser wavelength calibrations are within specification. The peak area ratio verification confirms that the probe intensity calibrations are within specification. The signal strength verification confirms that the instrument's signal-to-noise ratio is within specification. A report is generated showing the results of the verification steps along with a Pass/Fail indication.




This step is not required to collect a Raman spectrum, but it is highly recommended. Refer to the applicable probe or optics manual to determine the appropriate verification accessory, acceptable reference samples, and for information about how to verify the specific analyzer/probe combination.

8 Diagnostics and troubleshooting

Raman RunTime provides diagnostic information to help determine troubleshooting required on the analyzer. Refer to the System warnings and errors section of the *Raman RunTime Operating Instructions (BA02180C)* for additional information.

8.1 Warnings and errors

The **Status** button in the middle of the main view Status bar displays the current state of the system.

Symbol	Description
	When the system is fully calibrated and operating as expected, the Status button in the middle of the main view status bar reads OK and appears Green .
	If a system warning is encountered, the Status button changes to Yellow . Warnings should be acknowledged but immediate action may not be necessary. Click the Status button to view details of the warning. The most common warning occurs when all the channels are not occupied. The button pulses continuously until the problem is resolved. Click the Status button to view details about the warning.
	If a system error is encountered, the Status button changes to Red . An error requires immediate action to restore system performance. Click the Status button to view details about the error.

8.2 Contact information

For Technical Service, refer to our website (<https://endress.com/contact>) for the list of local sales channels in your area.

www.addresses.endress.com
