# Operating Instructions Accessory optics for the Rxn-10 probe KIO1, KNCO1, KLBIO1, KRSU1, KRBMO, KRBSL





# **Table of Contents**

1	About this document 3
1.1	Warnings3
1.2	U.S. export compliance3
1.3	Glossary4
2	Basic safety instructions
2.1	Requirements for the personnel5
2.2	Designated use5
2.3	Workplace safety6
2.4	Operational safety6
2.5	Service safety6
2.6	Important safeguards6
2.7	Product safety6
3	Product description7
3.1	Immersion optic (KIO1)7
3.2	Non-contact optic (KNCO1)9
3.3	bIO-Optic (KLBIO1) 10
3.4	Bio multi optic (KRBMO) and bio sleeve (KRBSL)
3.5	Raman optic system for single use (KRSU1) 12
4	Incoming product acceptance and product identification
4.1	Incoming acceptance 13
4.2	Product identification13
4.3	Scope of delivery13
5	Installation14
5.1	Installing immersion optics and bIO-Optics 14
5.2	Installing the bio multi optic and bio sleeve system

5.3	Installing non-contact optics19		
5.4	Installing the Raman optic system for single use		
6	Commissioning 24		
6.1	Receipt of optics24		
6.2	Calibration and verification24		
7	Operation25		
7.1	bIO-Optic, bio multi optic and bio sleeve system, and Raman optic system for single use25		
7.2	Storing the bio multi optic and the resusable portion of the Raman optic system for single use 		
8	Diagnostics and troubleshooting 27		
9	Maintenance 28		
9.1	Cleaning the optics window28		
9.2	Autoclaving the bIO-Optic28		
9.3	Autoclaving the bio sleeve		
10	Repair		
10.1	LRepairing optics for the Rxn-10 probe34		
10.2	2 User serviceable parts34		
11	Technical data 35		
11.1	I Immersion optic35		
11.2	2 Non-contact optic36		
11.3 bIO-Optic			
11.4	Bio multi optic and bio sleeve37		
11.5	Raman optic system for single use37		
12	Supplementary documentation 38		
13	Index 39		

# 1 About this document

This manual provides information about the optics used with the Endress+Hauser Rxn-10 Raman spectroscopic probe. The types of interchangeable optics available include:

- Immersion optic
- Non-contact optic
- bIO-Optic
- Bio multi optic and bio sleeve
- Raman optic system for single use

Refer to the *Rxn-10 Raman spectroscopic probe Operating Instructions* for specific probe-related information.

## 1.1 Warnings

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

Table 1. Warnings

## 1.2 U.S. export compliance

The policy of Endress+Hauser is in strict compliance with U.S. export control laws as detailed in the website of the Bureau of Industry and Security at the U.S. Department of Commerce.

# 1.3 Glossary

Term	Description
ANSI	American National Standards Institute
API	active pharmaceutical ingredient
bara	absolute pressure
barg	gauge pressure
°C	Celsius
cm	centimeter
°F	Fahrenheit
HCA	Raman Calibration Accessory
in.	inches
IO	immersion optic
IPA	isopropyl alcohol
kg	kilogram
LED	light emitting diode
m	meter
μin	microinches
μm	micrometer
mm	millimeter
mW	milliwatt
NCO	non-contact optic
NIR	near infrared region
nm	nanometer
PD	process development
psig	pounds per square inch gauge
Ra	roughness average
VIS	visible region
WEEE	Waste Electrical and Electronic Equipment

Table 2. Glossary

# 2 Basic safety instructions

The safety information in this section is specific to the optics that are compatible with the Rxn-10 Raman spectroscopic probe. Refer to the *Rxn-10 Raman spectroscopic probe Operating Instructions* for additional information related to probe and laser safety.

## 2.1 Requirements for the personnel

- Installation, commissioning, operation, and maintenance of the probe/optics 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.
- Technical personnel must have read and understood these Operating Instructions and must follow the instructions contained herein.
- The facility must designate a laser safety officer who ensures staff are trained on all Class 3B laser operating and safety procedures.
- Faults at the measuring point may only be rectified by authorized trained personnel. Repairs not described in this document must be carried out only at the manufacturer's site or by the service organization.

## 2.2 Designated use

The Rxn-10 Raman spectroscopic probe is designed for product and process development as well as manufacturing (when used with the Raman optic system for single use). The probe is compatible with a range of interchangeable, commercially available optics (immersion and non-contact) to meet the requirements of different applications.

Optic	Fields of application
Immersion optic (IO)	<ul> <li>Development laboratory</li> <li>Pharmaceutical: drug substance unit operations, reaction analysis, crystallization, end-point detection, solvent swaps</li> <li>Chemical: material identification, reaction analysis, polymerization, cross-linking, blending</li> <li>Food and beverage: blending, purification, natural and synthetic components</li> </ul>
Non-contact optic (NCO)	<ul> <li>Polymer solids (pellets, films, or powders)</li> <li>Pharmaceutical drug product manufacturing</li> <li>Raw material identification</li> <li>Meat or fish quality</li> <li>Formulation optimization</li> </ul>
bIO-Optic	<ul> <li>Benchtop bioreactors to measure glucose, lactate, amino acids, cell density, titer, and more</li> <li>Benchtop fermenters to measure glycerol, methanol, ethanol, sorbitol, biomass, and more</li> <li>Use with Flow assembly <u>CYA680</u> for select downstream bioprocessing applications</li> </ul>
Bio multi optic and bio sleeve	<ul> <li>Benchtop bioreactors to measure glucose, lactate, amino acids, cell density, titer, and more</li> <li>Benchtop fermenters to measure glycerol, methanol, ethanol, sorbitol, biomass, and more</li> <li>Use with Flow assembly <u>CYA680</u> for select downstream bioprocessing applications</li> </ul>
Raman optic system for single use	<ul> <li>Single-use bioreactors to measure glucose, lactate, amino acids, cell density, titer, and more</li> <li>Single-use fermenters to measure glycerol, methanol, ethanol, sorbitol, biomass, and more</li> </ul>

Recommended applications for the optics include:

Table 3. Fields of application

The above table lists common applications for the Rxn-10 probe and optics. There are other possible fields of application; however, use of the device for any purpose well outside of the fields of application described here poses a threat to the safety of people and of the entire measuring system and invalidates any warranty.

## 2.3 Workplace safety

As the user, you are responsible for complying with the following safety conditions:

- Installation guidelines
- Local standards and regulations for electromagnetic compatibility

## 2.4 Operational safety

Before commissioning the entire measuring point:

- Verify that all connections are correct.
- Ensure that electro-optical cables are undamaged.
- Ensure fluid level is sufficient for probe/optics immersion (if applicable).
- Do not operate damaged products, and protect them against unintentional operation.
- Label damaged products as defective.

During operation:

- If faults cannot be rectified, products must be taken out of service and protected against unintentional operation.
- When working with laser devices, always follow all local laser safety protocols which may include the use of
  personal protective equipment and limiting device access to authorized users.

Service ratings may include limitations for fittings, flanges, or seals. Installer must understand these limitations and use appropriate hardware and assembly procedures for a pressure-tight and safe joint.

## 2.5 Service safety

Follow your company's safety instructions when removing a process probe/optic from the process interface for service. Always wear proper protective equipment when servicing the equipment.

## 2.6 Important safeguards

- Do not use the optics for anything other than their intended use.
- Do not look directly into the laser beam.
- Do not point the laser at a mirrored/shiny surface or a surface that may cause diffuse reflections. The reflected beam is as harmful as the direct beam.
- When not in use, close the shutter on the Rxn-10 probe. If an optic cap is available, place it on the unused optic.
- Always use a laser beam block to avoid inadvertent scatter of laser radiation.

## 2.7 Product safety

The product is designed to meet all current safety requirements, has been tested, and shipped from the factory in a safe operating condition. The relevant regulations and international standards have been observed. Devices connected to an analyzer must comply with the applicable analyzer safety standards.

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# **3** Product description

The variety of optics available for the Rxn-10 Raman spectroscopic probe, powered by Kaiser Raman technology, enables flexible sampling options in the laboratory, process development, or single use manufacturing environment. Endress+Hauser offers immersion, bioprocess, and non-contact optics for the analysis of liquids, slurries and solids. Optics are available in multiple lengths and sizes to meet the requirements of different applications. Refer to the sections below for descriptions of the optics types and their use.

- 3.1: Immersion optic
- 3.2: Non-contact optic
- 3.3: bIO-Optic
- 3.4: Bio multi optic and bio sleeve
- 3.5: Raman optic system for single use

## 3.1 Immersion optic (KIO1)

The Endress+Hauser immersion optic is suited for use with the Rxn-10 probe in reaction vessels, laboratory reactors, or process streams. It has a fixed focus design and no movable parts, providing long-term measurement stability and superior signal performance. The sealed design is the standard for use with embedded Raman Rxn analyzers.







Figure 2. Tips of immersion optics with varying diameters

### 3.1.1 Benefits of the immersion optic

The immersion optic offers the following benefits for transferable, high-performance Raman measurements:

- Ideal for quick liquids, slurries and semisolids measurements in the laboratory
- Superior signal performance
- No user-adjustable parts
- Wettable components resist corrosion in harsh environments (including acidic solutions)

## 3.1.2 Immersion optic options

The immersion optic is available in 12.7 mm (0.5 in.) and 6.35 mm (0.25 in.) diameter configurations with two optical coating options:

- VIS: optimized for use in the visible (VIS) region (532 nm)
- NIR: optimized for use in the near infrared (NIR) region (785 nm and 993 nm)

#### 3.1.3 Data collection zone: short vs. long

The immersion optic can have either a short (at the window) or long (3 mm or 0.12 in. from the window) data collection zone. The data collection zone selected is also indicated on the immersion optic.

Short or long data collection zones are used for different kinds of samples. Spectral data is collected most efficiently at the focal plane.

A short data collection zone is generally used for opaque or turbid media samples. If an immersion optic with a long data collection zone were used to analyze these materials, most or all of the incident radiation would be lost to specular and diffuse reflection by material above the focal plane.

A long data collection zone is better for transparent samples because it maximizes the signal intensity by using the entire effective focal cylinder.



Figure 3. Short (left) vs. long (right) data collection zone (1)

## 3.2 Non-contact optic (KNCO1)

Paired with the Rxn-10 probe, the Endress+Hauser non-contact optic provides contact-free Raman measurements of samples either directly or through sight glass or translucent packaging. These optics are ideal for use with solids or turbid media or when sample contamination or damage to optical components is a concern.



Figure 4. Non-contact optics in varying sizes

## 3.2.1 Benefits of the non-contact optic

The non-contact optic offers the following benefits for Raman measurements:

- Range of working distances for remote measurements either directly or through sight glass and translucent packaging
- Highly versatile, measuring films to pellets to powders
- Accurate measurement of static or moving samples
- Contact-free analysis of delicate or corrosive samples

## 3.2.2 Non-contact optic options

Non-contact optics are available in a variety of sizes with a working distance range of 10 to 140 mm (0.40 to 5.52 in.) depending on the option selected. The internal lens comes with one of two types of anti-reflective coatings:

- VIS: optimized for use in the visible (VIS) region
- NIR: optimized for use in the near infrared (NIR) region

Refer to the table below for available options.

Non-contact optic size	Anti-reflective coating	Working distance (mm)	Working distance (in.)
NCO-0.4	NIR	10	0.40
NCO-0.5	VIS	12.5	0.50
NCO-1.3	VIS	33	1.30
NCO-2.5	VIS	64	2.52
NCO-3.0	NIR	75	2.96
NCO-5.5	VIS	140	5.52
NCO-5.5	NIR	140	5.52

Table 4. Non-contact optics

## 3.3 bIO-Optic (KLBIO1)

The Endress+Hauser bIO-Optic is a versatile immersion optic used in conjunction with the Rxn-10 probe. It measures multiple, specific bioprocessing components in real-time and is compatible with standard PG13.5 bioreactor ports. The fixed focus design of the bIO-Optic provides long-term measurement stability along with superior signal performance, essential for transferable, high performance Raman-based bioprocess analysis. Available in various industry standard lengths, the bIO-Optic is ideally suited for benchtop bioreactor/fermentor applications requiring headplate entry.



Figure 5. bIO-Optics in varying lengths

#### NOTICE

#### The bIO-Optic should NOT be used with hydrocarbon solvents (including ketones and aromatics).

• These solvents can degrade probe performance and invalidate the warranty.

## 3.3.1 Benefits of the bIO-Optic

The bIO-Optic offers the following benefits for *in situ* monitoring of bioprocess applications in process development (PD):

- Immersion optic for multi-component bioprocess measurement
- Compatible with standard PG13.5 bioreactor ports
- Fixed-focus design
- Available in various industry standard lengths
- Autoclavable; punch card included for tracking
- 120 mm version compatible with Endress+Hauser flow assembly <u>CYA680</u>

## 3.3.2 bIO-Optic options

The bIO-Optic is available in 120, 220, 320, or 420 mm (4.73, 8.67, 12.60, or 16.54 in.) lengths. The 12 mm (0.48 in.) diameter with PG13.5 threaded connector is ideally suited for headplate entry into the bioreactor/fermentor.

A bIO-Sample chamber is an available option that can be used for the probe verification procedure.



Figure 6. bIO-Optic with the bIO-Sample chamber

## 3.4 Bio multi optic (KRBMO) and bio sleeve (KRBSL)

The Endress+Hauser bio multi optic and bio sleeve make up a versatile, two part immersion optic system used in conjunction with the Rxn-10 probe. This system measures multiple, specific bioprocessing components in real-time and is compatible with standard PG13.5 bioreactor ports.

The system is comprised of the following parts:

- A reusable bioprocessing multi optic, which has no product contact, and
- The bioprocessing sleeve that interfaces with the bio multi optic and has product contact. The bio sleeve has a lifespan of 10 autoclave cycles when used in conjunction with the bio sleeve desiccator.



Figure 7. Bio multi optic and bio sleeve system in varying lengths

#### NOTICE

#### This system should NOT be used with hydrocarbon solvents (including ketones and aromatics).

• These solvents can degrade probe performance and invalidate the warranty.

## 3.4.1 Benefits of the bio multi optic and bio sleeve

The bio multi optic and bio sleeve system offers the following benefits:

- Modular design allows calibration of the optic without removing the bio sleeve from the bioreactor/wetted area.
- Reduced probe service and maintenance because of the simplification of the wetted/sterilized portion.
- Fixed focus design provides long-term measurement stability and superior signal performance, essential for transferable, high performance Raman-based bioprocess analysis.
- 120 mm version is compatible with Endress+Hauser flow assembly <u>CYA680</u>.

### 3.4.2 Bio multi optic and bio sleeve options

The bio multi optic and bio sleeve are available in 120 mm and 220 mm (4.73 in. and 8.67 in.) industry standard lengths. The sample system is ideally suited for benchtop bioreactor/fermentor applications requiring headplate entry.

A multi optic verification accessory is an available option that can be used for the probe verification procedure.

## 3.5 Raman optic system for single use (KRSU1)

The Endress+Hauser Raman optic system for single use was developed according to industry standards for single use sensors and is designed for single use bioprocess applications. The system is used in conjunction with the Rxn-10 probe and is comprised of the following parts:

- The reusable optic, which has no product contact, and
- A disposable fitting, which is installed, tested, and supplied ready to use from the single use vessel vendor.



Figure 8. Reusable optic (top) and disposable fitting (bottom)

#### NOTICE

The Raman optic system for single use should NOT be used with hydrocarbon solvents (including ketones and aromatics).

• These solvents can degrade probe performance and invalidate the warranty.

### 3.5.1 Benefits of the Raman optic system for single use

The Raman optic system for single use, in conjunction with the Rxn-10 probe, has been shown to offer the same quality data as reusable Raman probes. This ability allows customers to develop products and processes without limitations of the final production reactor type.

Additional benefits include:

- Disposable fitting plus a reusable non-contact optic
- Developed to industry standards for single use sensors
- cGMP qualified
- Gamma sterilizable
- Tested and supplied by multiple single use vessel vendors

Biopharmaceutical customers depend on its sterility and reliability for single use development and manufacturing.

## 3.5.2 Optic and fitting

The reusable optic is supplied by Endress+Hauser and does not break the sterile barrier.

The disposable fitting is purchased through the single use vessel vendor as an Endress+Hauser Raman-ready, single use vessel. The vessel is manufactured to the customer's specification with the fitting installed. The manufacturer tests and supplies a sterile single use product to the customer. The disposable fitting is intended to be used for a single batch cycle only.

# 4 Incoming product acceptance and product identification

## 4.1 Incoming acceptance

- 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.
- 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.
- Check that the delivery is complete and nothing is missing. Compare the shipping documents with your order.
- 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, please contact your supplier or your local sales center.

NOTICE

Incorrect transportation can damage the optics.

## 4.2 Product identification

## 4.2.1 Label

At a minimum, the optics are labeled with the following information:

- Manufacturer information
- Serial number

Compare the information on the label/tag with the order.

#### 4.2.2 Manufacturer address

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

## 4.3 Scope of delivery

The scope of delivery comprises:

- Selected optic(s)
- Accessory optics for the Rxn-10 probe Operating Instructions manual

If you have any questions, please contact your supplier or local sales center.

# 5 Installation

The installation information in this section is specific to the optics that are compatible with the Rxn-10 Raman spectroscopic probe. Refer to the *Rxn-10 Raman spectroscopic probe Operating Instructions* for additional information related to probe installation.

The Rxn-10 probe is compatible with both immersion optics and non-contact optics. The probe has a torque limiting clamp that secures the immersion optics. The clamp also holds the adapter for non-contact optics.

Before installation, ensure any protective covers are removed from the optics.

When replacing an optic in a probe, use the multi optic calibration accessory or the Raman Calibration Accessory (HCA) to perform an intensity calibration for that probe with the new optic. See Section  $11 \rightarrow \square$  to determine the appropriate calibration method for each optic.

## 5.1 Installing immersion optics and bIO-Optics

Endress+Hauser immersion optics and bIO-Optics slip into the Rxn-10 probe and are secured by a torque limiting, thumb screw-based clamp. The thumb screw on the Rxn-10 probe should never be fully removed.

#### 

# When installing or removing immersion optics, ensure the laser and emission shutter are in the closed position.

To install an immersion optic:

- 1. If necessary, loosen the metal thumb screw on the Rxn-10 probe by turning the screw counter-clockwise approximately one turn (do not remove). Then find the probe end of the optic, which is the end that includes the product markings.
- 2. Insert the probe end of the optic through the end optic clamp.
- 3. Push the optic back until it stops.
- 4. Tighten the thumb screw by gently turning it clockwise until there is an audible "click" sound. This indicates the thumb screw has reached the desired torque. Failure to tighten the screw will result in the optic coming loose, potentially damaging the optic.
- 5. After installing an optic into a probe, use the Raman Calibration Accessory to perform an intensity calibration for the probe with the new optic.



Figure 9. Installing an immersion optic (IO) on an Rxn-10 probe

To remove an immersion optic:

Loosen the torque limiting thumb screw by turning it counter-clockwise approximately one turn so that the immersion optic is released from its clamp. Do not remove the screw. Then slide the immersion optic out.

## 5.2 Installing the bio multi optic and bio sleeve system

Installation of the bio multi optic and bio sleeve system into a bioreactor for data acquisition is comprised of the following steps:

- Installing the bio multi optic into the Rxn-10 probe
- Installing the bio sleeve into the bioreactor
- Sterilizing the bio sleeve/bioreactor combination (typically)
- Installing the Rxn-10 probe with bio multi optic into the bio sleeve/bioreactor

#### 5.2.1 Installing the bio multi optic into the Rxn-10 probe

The Endress+Hauser bio multi optic slips into the Rxn-10 probe and is secured by a torque limiting, thumb screw-based clamp. The thumb screw on the Rxn-10 probe should never be fully removed.

#### **WARNING**

#### When installing or removing optics, ensure the laser and emission shutter are in the closed position.

To install the optic into the probe:

- 1. If necessary, loosen the metal thumb screw on the Rxn-10 probe by turning the screw counter-clockwise approximately one turn (do not remove).
- 2. Insert the optic through the end optic clamp.



Figure 10. Inserting the bio multi optic into the Rxn-10 probe

3. Push the optic back until it stops.



Figure 11. Final position of bio multi optic in the Rxn-10 probe

4. Tighten the thumb screw by gently turning it clockwise until there is an audible "click" sound. This indicates the thumb screw has reached the desired torque. Failure to tighten the screw will result in the optic coming loose, potentially damaging the optic.



Figure 12. Tightening the thumb screw on the Rxn-10 probe

5. After installing an optic into a probe, use the multi optic calibration accessory to perform an intensity calibration for the probe with the new optic. Alternately, the Raman Calibration Accessory (HCA) may be used, but a bio sleeve is required.

To remove the bio multi optic from the Rxn-10 probe:

Loosen the torque limiting thumb screw by turning it counter-clockwise approximately one turn so that the optic is released from its clamp. Do not remove the screw. Then slide the optic out.

### 5.2.2 Installing the bio sleeve into the bioreactor

The bio sleeve is designed to interface with a PG13.5 threaded port on the bioreactor. To install the bio sleeve into the bioreactor:

- 1. Align the bio sleeve with the available PG13.5 port.
- 2. Insert the bio sleeve into the PG13.5 port on the bioreactor.
- 3. Tighten the PG13.5 nut into the PG13.5 port of the bioreactor according to the bioreactor manufacturer's guidelines.

#### NOTICE

#### It is critical that there is no interference between the bio sleeve and the internal stirring device.

• Interference can damage the optic system and may cause cavitation.



Figure 13. Installing the bio sleeve into the PG13.5 port of the bioreactor

#### NOTICE

#### The bioreactor/bio sleeve will typically need to be sterilized prior to use.

• The desiccator must be installed into the bio sleeve prior to autoclave sterilization.

See Section  $9.3 \rightarrow \square$  for autoclaving instructions.

## 5.2.3 Installing the Rxn-10 probe with bio multi optic into the bio sleeve

#### NOTICE

#### Prior to insertion in the bio sleeve, the Rxn-10 probe with the reusable optic should be calibrated and verified.

• See Section 6.2  $\rightarrow \bigoplus$  for calibration and verification instructions.

After the Rxn-10 with bio multi optic is calibrated and the bioreactor with the installed bio sleeve is sterilized, the systems need to be joined to take data. Follow the steps below.

1. Obtain the sanitary flange seal.

If the sanitary flange seal is already present on the bio sleeve, go to step 3.



Figure 14. Sanitary flange seal

2. Place the sanitary flange seal onto the seal gland.



Figure 15. Installing the sanitary flange seal

- 3. Confirm that the bio multi optic for use is the correct immersion length.
- 4. Insert the bio multi optic into the bio sleeve until the optic rests on the sanitary flange seal.



Figure 16. Installing the bio multi optic into the bio sleeve (left) until the optic rests on the seal (right)

5. Confirm that the sanitary seal is properly seated between the optic and the sleeve.

6. Install the sanitary clamp, making sure it is clamped tightly. There should be two distinct clicks indicating it is clamped correctly.



Figure 17. Installing the sanitary clamp

The Rxn-10 probe with the bio multi optic is now ready to acquire data with the bio sleeve in the bioreactor.

## 5.3 Installing non-contact optics

The non-contact optics offered with the Rxn-10 probe are threaded, so a threaded adapter is required to attach the optic to the Rxn-10 probe.

#### **WARNING**

# When installing or removing non-contact optics, ensure the laser and emission shutter are in the closed position.

To install a non-contact optic:

- 1. If necessary, loosen the metal thumb screw on the Rxn-10 probe by turning the screw counter-clockwise approximately one turn (do not remove). Then find the narrow, non-threaded end of the adapter.
- 2. Insert the narrow end of the adapter through the clamp. Push the adapter back until it stops.
- 3. Tighten the thumb screw by gently turning it clockwise until there is an audible "click" sound. This indicates the thumb screw has reached the desired torque. Failure to tighten the screw will result in the adapter coming loose.
- 4. Find the externally threaded end of the non-contact optic.
- 5. Screw a non-contact optic into the threaded end of the adapter.
- 6. After installing an optic into a probe, use the Raman Calibration Accessory (HCA) to perform an intensity calibration for the probe with the new optic.



Figure 18. Installing an adapter and non-contact optic into the Rxn-10 probe

To remove a non-contact optic:

Unscrew the non-contact optic from the adapter. If an immersion optic will be used, remove the adapter by turning the torque limiting thumb screw counter-clockwise approximately one turn until the adapter is released from the clamp. Then slide the adapter out.

## 5.4 Installing the Raman optic system for single use

#### NOTICE

Prior to insertion in the disposable fitting, the Rxn-10 probe with the reusable optic should be calibrated and verified.

• See Section 6.2  $\rightarrow \cong$  for calibration and verification instructions.

## 5.4.1 Preparing the disposable fitting

The port shown below is specific to one type of single use bioreactor. The port, fitting, and cap/clamp (if present) may vary depending on the type of single use bioreactor. However, the optic insertion instructions are the same for all types of single use bioreactors.

To prepare the disposable fitting for optic insertion:

- 1. Depress the release lever on the sanitary clamp and remove the clamp.
- 2. Remove the sanitary cap from the fitting.
- 3. Ensure the sanitary o-ring seal is in place on the fitting.



Figure 19. Preparing the disposable fitting

## 5.4.2 Preparing the reusable optic for insertion into the fitting

To prepare the optic for insertion into the fitting:

- 1. Depress the release lever on the sanitary clamp and remove the clamp.
- 2. Remove the cap and seal. Store these components in a secure location.



Figure 20. Preparing the reusable optic

## 5.4.3 Inserting the optic in the fitting

To insert the optic in the disposable fitting:

1. Verify the sanitary o-ring seal on the fitting is still in place.

If not present, place a sanitary o-ring seal over the optic such that it is located in the sealing gland area.

- 2. Insert the optic into the disposable fitting.
- 3. Install the sanitary clamp, making sure it is clamped tightly. There should be two distinct clicks indicating it is clamped correctly.



Figure 21. Inserting the optic in the fitting

### 5.4.4 Installing the optic into the Rxn-10 probe

The Endress+Hauser reusable optic slips into the Rxn-10 probe and is secured by a torque limiting, thumb screwbased clamp. The thumb screw on the Rxn-10 probe should never be fully removed.

#### **WARNING**

#### When installing or removing optics, ensure the laser and emission shutter are in the closed position.

To install the optic into the probe:

- 1. If necessary, loosen the metal thumb screw on the Rxn-10 probe by turning the screw counter-clockwise approximately one turn (do not remove). Then insert the optic through the end optic clamp.
- 2. Push the optic back until it stops.
- 3. Tighten the thumb screw by gently turning it clockwise until there is an audible "click" sound. This indicates the thumb screw has reached the desired torque. Failure to tighten the screw will result in the optic coming loose, potentially damaging the optic.



Figure 22. Installing the reusable optic into the Rxn-10 probe

4. After installing an optic into a probe, and before it is connected to the fitting, use the multi optic calibration accessory to perform an intensity calibration for the probe with the new optic. Alternately, the Raman Calibration Accessory (HCA) and single use calibration adapter may be used.

To remove the optic:

Loosen the torque limiting thumb screw by turning it counter-clockwise approximately one turn so that the optic is released from its clamp. Do not remove the screw. Then slide the optic out.

# 6 Commissioning

The optics for the Rxn-10 probe are delivered ready to connect to the probe. No additional alignment or adjustment to the probe head is required. Follow the instructions below to commission the optics for use in conjunction with the probe.

## 6.1 Receipt of optics

Perform the steps for incoming product acceptance described in Section  $4.1 \rightarrow \square$ .

## 6.2 Calibration and verification

The probe and the analyzer must be calibrated before use.

## 6.2.1 Multi optic calibration accessory

After installing the bio multi optic or the Raman optic system for single use into the Rxn-10 probe, use the multi optic calibration accessory to perform an intensity calibration for the probe head with the new optic.

Refer to the *Multi optic calibration kit Operating Instructions* for additional information about the multi optic calibration accessory.

If the multi optic calibration accessory is not available, a Raman Calibration Accessory (HCA) may be used for calibration as follows:

- Bio multi optic: with bio sleeve and 12 mm HCA adapter
- Raman optic system for single use: with the single use calibration adapter and 12 mm HCA adapter

## 6.2.2 Raman Calibration Accessory

After installing an immersion optic, non-contact optic, or bIO-Optic into the probe head, use the Raman Calibration Accessory (HCA) to perform an intensity calibration for the probe head with the new optic.

If the HCA is used with the Raman optic system for single use, an additional single use calibration adapter is installed onto the optic following the same process as connecting the optic to the fitting (see Section  $5.3 \rightarrow \textcircled{}$ ). The optic/calibration adapter combination is then inserted into an HCA adapter attached to the HCA head.

Refer to the *Raman Calibration Accessory Operating Instructions* for additional information about the HCA and adapters.

## 6.2.3 Performing calibration and verification

Refer to the applicable Raman Rxn analyzer operating instructions for steps to:

- Perform internal analyzer calibration; may include alignment calibration, full wavelength calibration and/or full laser wavelength calibration depending on status of analyzer
- Perform probe calibration; requires multi optic calibration accessory or HCA with an appropriate optic adapter
- Perform probe verification; verifies the calibration results using a standard reference sample; a bIO Sample Chamber or multi optic verification accessory may be used
- View calibration and verification reports

#### NOTICE

The multi optic verification accessory should be used for verification of the bio multi optic or Raman optic system for single use. Do NOT immerse the bio multi optic or optic for single use directly into a sample.

If the multi optic verification accessory is not available, verification of the bio multi optic or Raman optic system for single use may be performed using a bIO Sample Chamber and an additional bio sleeve (for bio multi optic) or a single use calibration adapter (for single use optic).

The Raman RunTime software will not allow spectra to be collected without passing internal and probe calibrations. Passing the probe verification step is not required but highly recommended.

Raman Rxn analyzer operating instructions are available by searching the Downloads area of the Endress+Hauser web site: <u>https://endress.com/downloads</u>

# 7 Operation

This manual provides information about the optics used with the Endress+Hauser Rxn-10 Raman spectroscopic probe. The Rxn-10 probe is a versatile probe designed for product and process development and is compatible with Endress+Hauser Raman Rxn analyzers operating at 532 nm, 785 nm, or 993 nm. The Rxn-10 probe accepts a variety of interchangeable optics including:

- Immersion optic
- Non-contact optic
- bIO-Optic
- Bio multi optic and bio sleeve
- Raman optic system for single use

The optics are installed into the probe following the instructions in Section  $5 \rightarrow \square$ .

Refer to the *Rxn-10 Raman spectroscopic probe Operating Instructions* for operation of the probe with the optics. Standard precautions for laser products should be observed.

Additional use and storage instructions for some optics are provided below.

# 7.1 bIO-Optic, bio multi optic and bio sleeve system, and Raman optic system for single use

The bIO-Optic, the bio multi optic and bio sleeve system, and the Raman optic system for single use should NOT be used with hydrocarbon solvents (including ketones and aromatics). Doing so can degrade probe performance and invalidate the warranty.

The Raman optic system for single use is NOT intended to be submerged into any liquid without being attached to the disposable fitting. The bio multi optic is NOT intended to be submerged into any liquid without being attached to a bio sleeve.

# 7.2 Storing the bio multi optic and the resusable portion of the Raman optic system for single use

When storing the bio multi optic or the reusable portion of the Raman optic system for single use, it is important to always keep the optic protected with the cover provided during shipping. Verify the o-ring seal is installed to ensure a clean and dry environment.



Figure 23. The cover for the reusable optic with o-ring present (left) and o-ring and cover for the bio multi optic (right)

#### 

#### When installing or removing optics, ensure the laser and emission shutter are in the closed position.

To remove the optic and install the optic cover for storage:

- 1. Loosen the metal thumb screw on the Rxn-10 probe by turning the screw counter-clockwise approximately one turn (do not remove).
- 2. Slide the Rxn-10 probe head off the bio multi optic or reusable optic.
- 3. Locate the quick release clamp, the optic cover, and the sanitary seal (o-ring).
- 4. Slide the optic into the cover, with the o-ring creating a seal between the two pieces.
- 5. Install the quick release clamp over the optic/cover flange and squeeze the clamp until you hear two distinct clicks to tighten.



Figure 24. Installing an optic cover and clamp

# 8 Diagnostics and troubleshooting

Refer to the *Rxn-10 Raman spectroscopic probe Operating Instructions* to troubleshoot issues with the Rxn-10 probe and accessory optics.

## 9 Maintenance

If the optics have come in contact with a sample, dust or fingerprints, etc., they may need to be cleaned. Additionally, the bIO-Optic and bio sleeve should be cleaned after immersion in phosphate buffer solutions to avoid particle deposit contamination.

The window on each of the optics can be cleaned following the steps below. For the bIO-Optic and bio sleeve, sterilization by autoclaving is typically performed.

For all other maintenance, it is recommended that the optics are serviced at the manufacturer's site.

## 9.1 Cleaning the optics window

Extra care must be taken to ensure that the window surface is not further contaminated during the cleaning process.

#### NOTICE

# Do NOT use hydrocarbon solvents (including ketones and aromatics) with the bIO-Optic, bio multi optic and bio sleeve system, or Raman optic system for single use.

• These solvents can degrade probe performance and invalidate the warranty.

To clean the optics window:

- 1. Ensure that the laser is turned OFF or the probe is disconnected from the analyzer.
- 2. Blow off the surface with clean compressed air to remove any loose particles.
- 3. Wipe the surface using a swab lightly dampened with a solvent appropriate for the substance to be cleaned. Solvents may include 100 % isopropyl alcohol (IPA), deionized water, or others.

Do not allow the solvent to drip behind the retaining components.

- 4. Wipe the surface dry with a dry swab.
- 5. Repeat the cleaning with an additional solvent, if needed, and wipe the surface dry with a dry swab.
- 6. Blow with clean compressed air to remove any swab remnants.
- 7. Inspect the surface to verify the effectiveness of the cleaning.

Verification with an inspection microscope in the cleaning process is highly recommended to look for smeared contaminants, swab remnants, etc., that may cause increased spectrum background.

8. Repeat the previous steps as necessary.

## 9.2 Autoclaving the bIO-Optic

The bIO-Optic is rated for 25 autoclave cycles at 131 °C (268 °F) when used with the autoclave end cap. After that, the bIO-Optic must be replaced or returned for service. Contact your local Endress+Hauser service provider for additional information.

## 9.2.1 Preparing for autoclaving

To prepare the bIO-Optic for autoclaving:

- 1. Close the shutter on the Rxn-10 probe.
- 2. Use the key on the front of the Raman Rxn analyzer to turn OFF the laser power. Use a laser safety card to ensure the laser is not emitting from any probes during this step.
- 3. Loosen the metal thumb screw on the Rxn-10 probe by turning the screw counter-clockwise approximately one turn (do not remove).
- 4. Carefully disconnect the bIO-Optic from the probe.
- 5. Store the Rxn-10 probe in a safe location (away from traffic, heat, etc.).

- 6. Clean the bIO-Optic probe tip and window:
  - $\circ$  Spray with reagent alcohol/70 % IPA.
  - Wipe gently with a lint-free wipe.
  - Verify that no lint remains on the optic after cleaning.
  - Make sure that the optic is dry before proceeding.
- 7. Insert the bIO-Optic into a bioreactor and tighten the connection adaptor.
- 8. Attach the autoclave end cap to the rear of the bIO-Optic outside the bioreactor following the steps in Section  $9.2.2 \rightarrow \square$ .

### 9.2.2 Installing and removing the autoclave end cap

Each bIO-Optic includes an end cap that must be installed before the bIO-Optic can be autoclaved.



Figure 25. bIO-Optic with end cap and autoclave recording punch card

To install the autoclave end cap and track autoclave cycles:

- 1. Insert the autoclave end cap onto the rear end of the bIO-Optic. The rear end of the optic contains the product markings and is slightly larger in diameter.
- 2. Push the end cap all the way down onto the bIO-Optic until a faint snap is heard, indicating the end cap is securely fastened onto the bIO-Optic.
- 3. On the autoclave recording punch card, punch out the next number on the card and discard the scrap punch piece. Note:
  - It is advisable to punch out the next cycle count prior to autoclaving to avoid handling the card when it is hot.
  - The autoclave recording punch card can remain attached to the bIO-Optic during autoclaving.
  - If the punch card is not available, use an alternate method to track the number of autoclave cycles for the bIO-Optic.
  - The bIO-Optic is now ready for autoclaving.



Figure 26. Using the autoclave recording punch card

To remove the autoclave end cap:

- 1. Push down on the top of the autoclave end cap.
- 2. Press in the release ring.
- 3. Remove the end cap. It should slide off easily.

## 9.3 Autoclaving the bio sleeve

The bio sleeve is rated for 10 autoclave cycles at 131 °C (268 °F) with the use of the bio sleeve desiccator. After 10 autoclave cycles, the bio sleeve should be replaced.

### 9.3.1 Preparing the sleeve desiccator

The bio sleeve should be sterilized prior to use. A sleeve desiccator with new desiccant must be installed into the bio sleeve for the sterilization process. Follow the steps below to replace the desiccant on the sleeve desiccator.

- 1. Confirm that the desiccator is the correct length.
- 2. Remove any existing desiccant (if present) by grasping the desiccant and pulling it toward the end of the desiccator shaft.



Figure 27. Removing desiccant from the sleeve desiccator

- 3. Discard the used desiccant and open the package containing the new desiccant. Only use desiccant from a freshly opened package.
- 4. Install the new desiccant by sliding it over the end of the desiccator until it reaches the shoulder. You should hear a faint click once it passes the retaining ring.



Figure 28. Installing desiccant on the sleeve desiccator

## 9.3.2 Installing the desiccator into the bio sleeve

The desiccator must be installed before autoclaving the bio sleeve.

1. Place the sanitary seal provided with the desiccator onto the gland on the bio sleeve.



Figure 29. Placing the sanitary seal onto the bio sleeve gland

2. Slide the desiccator with new desiccant into the bio sleeve to be autoclaved, ensuring that the gland aligns with the seal.



Figure 30. Inserting the desiccator with desiccant (left) until the desiccator is aligned with the bio sleeve (right)

3. Install the sanitary clamp, making sure it is clamped tightly. There should be two distinct clicks indicating it is clamped correctly.

The bio sleeve is now ready for sterilization.



Figure 31. Sanitary clamp connecting desiccator and bio sleeve

## 9.3.3 Removing the desiccator from the bio sleeve

After sterilization, the desiccator must be removed from the bio sleeve before installing the bio multi optic. To remove the desiccator:

- 1. Depress the release lever on the sanitary clamp and remove the clamp.
- 2. Slide the desiccator out of the bio sleeve.
- 3. Leave the sanitary flange seal in place for installation of the Rxn-10 probe with bio multi optic. See Section 5.2.3  $\rightarrow \square$  for installation instructions.
- 4. Store the clamp and desiccator in a secure location.

# 10 Repair

## 10.1 Repairing optics for the Rxn-10 probe

Repairs not described in this document must be carried out only directly at the manufacturer's site or by the service organization. For Technical Service, refer to our website (https://endress.com/contact) for the list of local sales channels in your area.

If a product must be returned for repair or replacement, follow all decontamination procedures indicated by your service provider.

### **WARNING**

#### Failure to properly decontaminate wetted parts before return can result in a fatal or serious injury.

To ensure swift, safe and professional product returns, please contact your service organization.

For additional product return information, refer to the following site and select the applicable market/region: https://www.endress.com/en/instrumentation-services/instrumentation-repair

## **10.2** User serviceable parts

For product and spare parts ordering information, please visit <u>www.endress.com</u> or contact your local sales center.

There are no user serviceable parts for the following:

- Immersion optic
- Non-contact optic
- bIO-Optic

# 11 Technical data

The specifications for the Rxn-10 probe in conjunction with each of the optics are listed in the tables below. Additionally:

- Maximum pressure for the immersion optic and bIO-Optic is calculated per ASME B31.3 2020 Edition for material and probe geometry at temperatures not to exceed the maximum listed.
- Minimum pressure rating: All probes have a minimum pressure rating of 0 bara (full vacuum). However, unless
  specified, they are not rated for low outgassing at high vacuum service.

## 11.1 Immersion optic

Item		Description		
Laser wavelength		532 nm, 785 nm, 993 nm		
Spectral coverage		limited by the coverag	e of the analyzer being us	ed
Maximum laser powe	er into probe head	< 499 mW		
Relative humidity		sealed:	up to 95 %, non-conder	ising
		non-sealed:	non-sealed: 20 to 60 %, non-condensing	
Sample interface	temperature	316L stainless steel:	–30 to 120 °C	(–22 to 248 °F)
		C276 alloy:	–30 to 280 °C	(−22 to 536 °F)
		Grade 2 titanium:	−30 to 315 °C	(−22 to 599 °F)
	maximum	316L stainless steel:	142.4 barg	(2066 psig)
	pressure <sup>1</sup> ,	C276 alloy:	158.1 barg	(2293 psig)
	12.7 mm (0.5 in.) diameter IO	Grade 2 titanium:	65.2 barg	(946 psig)
	maximum pressure <sup>1</sup> , 6.35 mm (0.25 in.) diameter IO	316L stainless steel:	168.5 barg	(2444 psig)
		C276 alloy:	186.2 barg	(2701 psig)
		Grade 2 titanium:	76.3 barg	(1107 psig)
Wetted materials	metal	C276 alloy standard		
		316L stainless steel or Grade 2 titanium upon request		
	window	high-purity sapphire, proprietary compression fit non-brazed design		
Shaft length	12.7 mm (0.5 in.) diameter IO	152 mm	(6 in.)	
		305 mm	(12 in.)	
		457 mm	(18 in.)	
	6.35 mm (0.25 in.) diameter IO	152 mm	(6 in.)	
		203 mm	(8 in.)	
Working distance	short (S)	0 mm	(0 in.)	
	long (L)	3 mm	(0.12 in.)	
Calibration method	532 nm	HCA-532		
	785 nm	HCA-785		
	993 nm	HCA-1000		
Verification method	532 nm	Immerse in cyclohexar	16	
	785 nm, 993 nm	Immerse in cyclohexane or 70 % IPA		

Table 5. Immersion optic specifications

<sup>&</sup>lt;sup>1</sup> Maximum service pressure ratings do not include the ratings of any fittings or flanges used to mount the probe into the process system. These items need to be independently evaluated and may lower the maximum service pressure of the probe.

# 11.2 Non-contact optic

Item		Description
Laser wavelength		532 nm, 785 nm, 993 nm
Spectral coverage		limited by the coverage of the analyzer being used
Maximum laser powe	er into probe head	< 499 mW
Sample interface	temperature	ambient
	pressure	ambient
	relative humidity	ambient
Wetted materials		optic dependent
Length		varies based on model
Diameter		varies based on model
Working distance		10 to 140 mm (0.40 to 5.52 in.), depending on optic
		see Section 3.2.2 $\rightarrow$
Calibration method	532 nm	HCA-532
	785 nm	HCA-785
	993 nm	HCA-1000
Verification method	532 nm	cyclohexane cuvette
	785 nm, 993 nm	cyclohexane or 70 % IPA cuvette

Table 6. Non-contact optic specifications

# 11.3 bIO-Optic

Item		Description		
Laser wavelength		785 nm, 993 nm		
Spectral coverage		limited by the coverage o	f the analyzer being used	
Maximum laser pow	er into probe head	< 499 mW		
Sample interface	temperature	–30 to 150 °C	(−22 to 302 °F)	
	maximum pressure	13.8 barg	(200 psig)	
Wetted materials	body	316L stainless steel		
	window	proprietary material, opti	imized for bioprocesses	
	process connection	PG13.5		
	surface finish	Ra 0.38 µm (Ra 15 µin) with electropolish		
	adhesive	USP Class VI and ISO 109	93 compatible	
Immersible length		120 mm	(4.73 in.)	
		220 mm	(8.67 in.)	
		320 mm	(12.60 in.)	
		420 mm	(16.54 in.)	
Immersible diameter		12 mm	(0.48 in.)	
Sterilization method		autoclave		
		rated for 25 autoclave cy	cles (30 minutes each) at 131 °C (268 °F)	
Calibration	785 nm	HCA-785		
method	993 nm	HCA-1000		
Verification method	785 nm, 993 nm	bIO Sample Chamber with	h 70 % IPA	

Table 7. bIO-Optic specifications

# 11.4 Bio multi optic and bio sleeve

Item		Description		
Laser wavelength		785 nm		
Spectral coverage		limited by the coverage of the analyzer being used		
Maximum laser pow	er into probe head	< 499 mW		
Sample interface	temperature	-30 to 150 °C (-22 to 302 °F)		
	maximum pressure	13.8 barg (200 psig)		
Wetted materials	body	316L stainless steel		
(bio sleeve)	window	proprietary material, optimized for bioprocesses		
	process connection	PG13.5		
	surface finish	Ra 0.38 µm (Ra 15 µin) with electropolish		
	adhesive	USP Class VI and ISO 10993 compatible		
Immersible length (bio sleeve)		120 mm (4.73 in.)		
		220 mm (8.67 in.)		
Immersible diameter	r (bio sleeve)	12 mm (0.48 in.)		
Sterilization method (bio sleeve)		autoclave (with the use of the bio sleeve desiccator) rated for 10 autoclave cycles (30 minutes each) at 131 $^{\circ}$ C (268 $^{\circ}$ F)		
Calibration method	785 nm	multi optic calibration accessory (recommended) or HCA-785 with bio sleeve attached to bio multi optic		
Verification method	785 nm	multi optic verification accessory with 70 % IPA (recommended) or bIO Sample Chamber with 70 % IPA and bio sleeve attached to bio multi optic		

Table 8. Bio multi optic and bio sleeve specifications

## **11.5** Raman optic system for single use

Item		Description		
Laser wavelength		785 nm, 993 nm		
Spectral coverage		limited by the coverage of the analyzer being used		
Maximum laser power into probe head		< 499 mW		
Sample interface temperature		0 to 100 °C (32 to 212 °F)		
Immersible length		dimensions vary according to single use bioreactor vendor port and fitting type		
Immersible diameter		dimensions vary according to single use bioreactor vendor port and fitting type		
Calibration	785 nm	multi optic calibration accessory (recommended) or		
memou	993 nm	HCA-1000 with single use calibration adapter		
Verification method785 nmmulti optic verification accord bIO Sample Chamber with		multi optic verification accessory with 70 % IPA (recommended) or bIO Sample Chamber with 70 % IPA and single use calibration adapter		
	993 nm	bIO Sample Chamber with 70 % IPA and single use calibration adapter		

Table 9. Raman optic system for single use specifications

# 12 Supplementary documentation

All documentation is available:

- On the Endress+Hauser Operations App for smartphone/tablet
- In the Downloads area of the Endress+Hauser website: https://endress.com/downloads

Part number	Document type	Document title
KA01551C	Brief Operating Instructions	Accessory optics for the Rxn-10 probe Brief Operating Instructions
TI01635C	Technical Information	Accessory optics for the Rxn-10 probe Technical Information

Table 10. Supplementary documentation

## 13 Index

adapters single use calibration, 23, 24 threaded, 19 data collection zone, 8 export compliance, 3 glossary, 4 optics additional documents, 38 cleaning, 28 designated use, 5 installation, 5, 14 operation, 25 receipt, 13 sterilizing, 28, 30 storage, 25 troubleshooting, 27 wetted materials, 35, 36, 37 probe calibration, 14, 17, 19, 21, 23, 24, 35, 36, 37

clamp, 14 verification, 10, 17, 21, 24, 35, 36, 37 Raman RunTime, 24 repair, 34 safety, 6 basic, 5 operational, 6 product, 6 service, 6 workplace, 6 specifications diameter, 10, 36, 37 humidity, 35, 36 laser power, 35, 36, 37 length, 35, 36, 37 pressure, 35, 36, 37 temperature, 35, 36, 37 working distance, 35, 36 technical data, 35

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