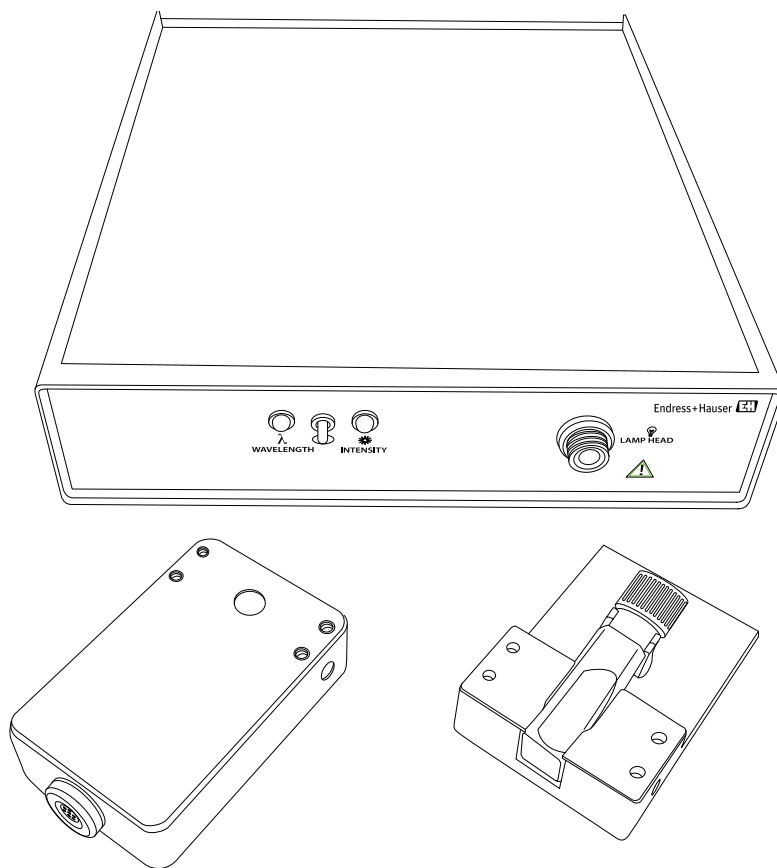


# Operating Instructions

## Raman calibration accessory








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# 1 About this document

## 1.1 Warnings

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

## 1.2 Symbols on the device

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 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 in the website of the [Bureau of Industry and Security](#) at the U.S. Department of Commerce.

## 1.4 Glossary

Term	Description
CCD	Charge coupled device
cm	Centimeter
DC	Direct current
FC	Ferrule connector
EEA	European economic area
HCA	Raman calibration accessory
HPLC	High performance liquid chromatography
Hz	Hertz
IEC	<a href="#">International Electrotechnical Commission</a>
kg	Kilogram
IO	Immersion optic
lbs	Pounds
LED	Light emitting diode
mm	Millimeter
NCO	Non-contact optic
NIST	<a href="#">National Institute of Standards and Technology</a>
nm	Nanometer
NMR	Nuclear magnetic resonance
SMA	Subminiature assembly
USB	Universal serial bus
W	Watt
WEEE	<a href="#">Waste Electrical and Electronic Equipment</a>

## 2 Basic safety instruction

### NOTICE

- ▶ The safety information in this section is specific to the Raman calibration accessory. Refer to the *Raman Rxn2 (BA02151C)*, *Raman Rxn4 (BA02178C)*, and *Raman Rxn5 (BA02179C) Operating Instructions* for additional analyzer-related safety information about working with lasers.

### 2.1 Designated use

The Calibration Accessory is used for standardizing Raman instruments and analyzers to give precise spectra in terms of intensity.

Use of the Calibration Accessory for any purpose other than that described poses a threat to the safety of people and of the entire measuring system and is not permitted.

### 2.2 Electrical safety

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

- Installation guidelines
- Local standards and regulations electromagnetic compatibility

### 2.3 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 undamaged.
3. Do not operate damaged products, and protect them against unintentional operation.
4. Label damaged products as defective.

### NOTICE

- ▶ During operation, if faults cannot be rectified products must be taken out of service and protected against unintentional operation.

### 2.4 Product safety

The Raman calibration accessory is designed to meet safety requirements, has been tested, and left the factory in a condition in which it is safe to operate. The relevant regulations and international standards have been observed. Devices connected to Raman Rxn analyzers must comply with the applicable safety standards.

### 2.5 Important safeguards

- Do not use the Raman calibration accessory for anything other than its intended use.
- Do not drape the power cord over counters or on hot surfaces.
- Do not open the enclosure of the Raman calibration accessory.
- Do not look directly into the laser beam.
- Do not stare at diffused or reflected laser light.
- Do not point a laser at a mirrored surface.
- Do not leave attached and unused probes uncapped or unblocked.
- Avoid shiny surfaces and always use a laser beam block.

### 2.6 Health and safety considerations

It is the user's responsibility to understand and comply with all applicable safety regulations. Safety regulations vary based on the installation location of the instrument. Endress+Hauser takes no responsibility for determining the safe use of the instrument based on this qualification procedure.

### 3 Product description

The Raman calibration accessory is used for standardizing Raman instruments and analyzers in terms of radiometric intensity. When used in conjunction with the calibration protocol recommended in this manual, the Raman calibration accessory allows different instruments to be standardized so that they generate similar spectra when measuring a given sample. The Raman calibration accessory was created specifically for use with Raman instruments and analyzers manufactured by Endress+Hauser.

The Raman calibration accessory contains an intensity reference lamp housed in a compact lamp head. The lamp head is connected to the control unit by a 1.8 m (6 ft) cable with positive locking, quick connectors at both ends. A diffuser window on the lamp head emits a quasi-lambertian pattern of light that, when properly positioned, fills the numerical aperture of a probe lens, microscope objective, or optical fiber.

For intensity standardization, a long-life, low-voltage tungsten-halogen lamp provides a factory-characterized spectral output. The primary reference source used in the certification process is a [National Institute of Standards and Technology](#) (NIST) traceable source. The halogen cycle maintains near constant color temperature throughout the operating life of the lamp under constant current operation. A precision current-regulated power source in the control unit assures consistent spectral output over many hours of operation.

#### NOTICE

- ▶ The Raman Rxn2 and Raman Rxn4 analyzers have built-in wavelength axis and laser wavelength calibration. The Raman calibration accessory should not be used for these kinds of calibrations on Raman Rxn2 and Rxn4 analyzers.

Analyzer	Intensity Axis	Verification
Raman Rxn2	✓	✓
Raman Rxn4	✓	✓

Table 1. Raman calibration accessory and Raman Analyzer compatibility


A GRAMS data file (.spc format) characterizing the spectral output of the lamp head (intensity mode) is provided on physical media. This file is referred to as the source spectral file (SSF). Because the SSF is relative, use of this accessory corrects only the normalized shape of measured spectra, not the absolute magnitude. The software provided with your analyzer is configured to reference the accessory's SSF in the instrument standardization process.

Instrument intensity standardization corrects for instrument-to-instrument response variations:

- Fixed-pattern noise caused by pixel-to-pixel variations in the response of individual charge-coupled device (CCD) detectors
- Spectral variations in signal intensity due to grating and lens transmission
- Spectral variations in the quantum efficiency of the CCD camera

Standardization refers to the process of using radiometric means to normalize individual analyzers so that spectral, and hence chemical, models can be transferred to multiple analyzers.

The lamp head and Raman shift standard vial are both sized to be interfaced by a microscope slide holder for the RamanRxn systems family of instruments. Optional adapters allow direct mechanical interface of the lamp head and Raman shift standard vial holder to regular Endress+Hauser probe head optics.

To maximize the life of the accessory, the halogen lamp automatically turns off after 45 minutes of uninterrupted "on" time. You can turn the lamp off manually at any time. The control unit keeps track of cumulative burn time on the halogen lamp since calibration. You are warned of both the approach and the passing of the 500-hour recommended replacement and re-calibration cycle as detailed in *Halogen lamp on/off* → .

Following standardization, Raman RunTime allows for system verification to be automatically performed with one of three Raman shift standards. Customers may also choose to do their own verification with other samples offline. However, a valid Raman RunTime verification using one of the three approved Raman shift standards is needed for the analyzer software to operate and collect data.

A certificate and spectral file accompanies each unit when new and when recertified.

#### NOTICE

- ▶ Endress+Hauser recommends recertification after 500 hours or 12 months (whichever comes first).

### 3.1 Controls and connections

The figure below shows a front view of the Raman calibration accessory, including the control unit and the lamp head. The front of the control unit contains the lamp power control switch, the wavelength (neon) indicator light emitting diode (LED) to the left of the switch, the intensity (halogen) indicator LED to the right of the switch, and the lamp head socket. The lamp head has a similar connector but with a reverse gender.

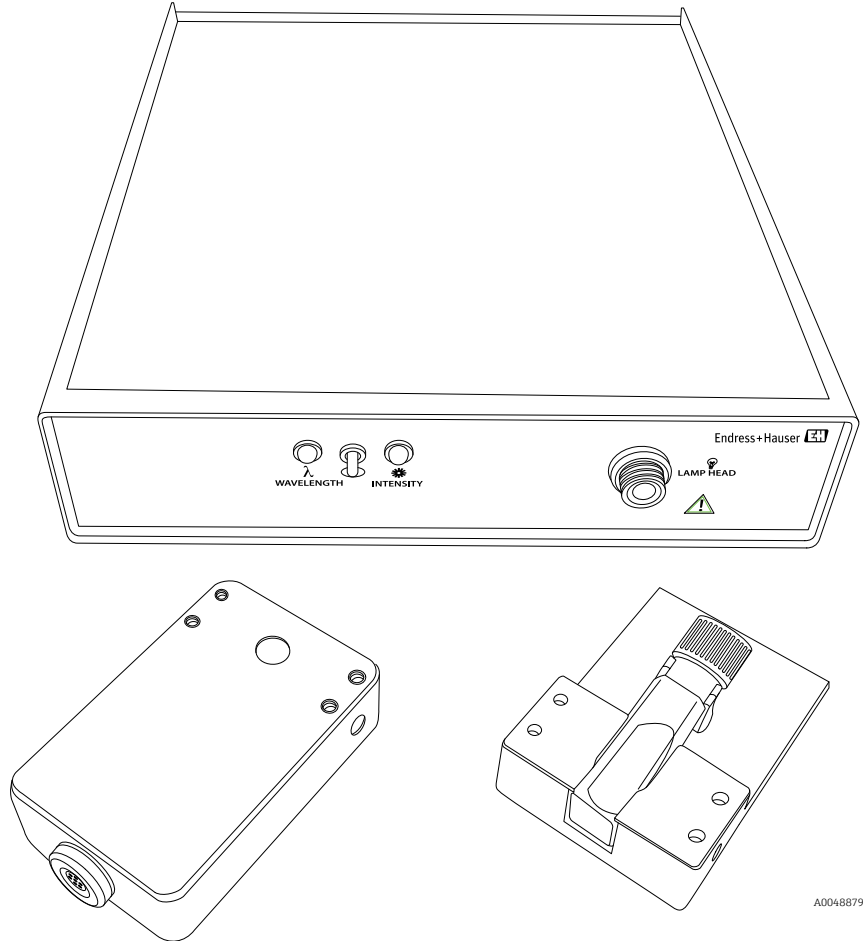


Figure 1: Front view of the control unit with the lamp head

The figure below is a rear view of the control unit, showing the power connection, the unit power ON/OFF switch, the fuse drawer and a universal serial bus (USB) communications port.

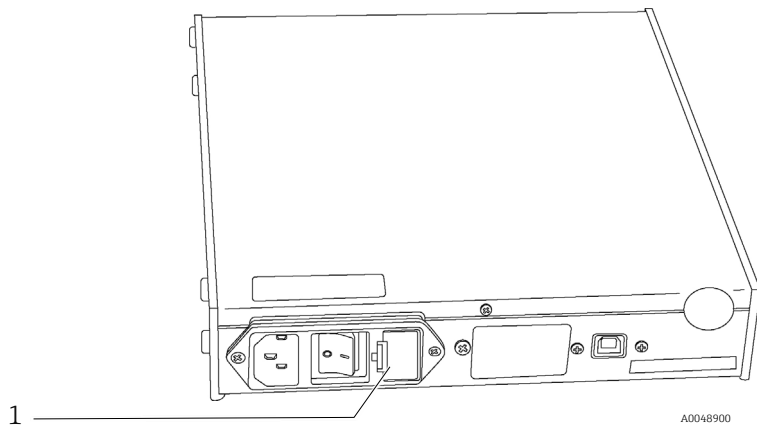


Figure 2: Rear view of the control unit with fuse drawer (1)



**NOTICE**

**Do not attempt communication. Damage could occur.**

- ▶ The USB port is for factory use only. The communication protocol is proprietary.

### 3.1.1 Unit power on/off

The switch on the power entry module at the back of the control unit activates power to the unit.

### 3.1.2 Lamp head cable/connector

The 1.8 m (6 ft) cable connecting the lamp head and the control unit has opposing connectors on both ends. The red dot on the cable plugs must be aligned with the red dot on the sockets for mating. The connector locks when mated. Pulling directly on the spring-loaded connector body releases the connection.

**⚠ CAUTION**

**Hazardous live voltages exist at the lamp head.**

- ▶ There are no user-serviceable parts in the lamp head. Do not operate this equipment with the lamp head unplugged from the control unit.

### 3.1.3 Halogen lamp on/off

Toggling the front panel switch to the right turns on the halogen intensity lamp and lights the right green LED. The lamp will turn off automatically after 45 minutes. The base unit keeps track of the lamp's elapsed "on" time (to the nearest 0.1 minute). When the lamp time exceeds 450 hours, the LED indicator lights yellow. When lamp time exceeds 500 hours, the LED indicator lights red. When the LED lights yellow or red, return the unit to Endress+Hauser for recertification.

Allow the halogen lamp be allowed to warm up for 12.5 minutes for the color temperature of the bulb to stabilize prior to its use. The halogen lamp LED indicator as a visual indicator of bulb warm-up time and lights solid when the warm-up period is complete.

The halogen lamp can be turned off by toggling the front panel switch to the right again. Toggling the switch to the left lights the neon lamps.

### 3.1.4 Power

The Raman calibration accessory uses a universal input switching power supply and will operate over a line input range of AC 100 to 240 V, 50 to 60 Hz. Main (line) power connection is through a standard power cord with universal IEC320 connector. Power consumption is 30 (W) maximum.

### 3.1.5 Fuses

Fuses are replaceable through a drawer next to the power switch on the back of the control unit. Always replace with two each AC 250 V-rated metric (5x20 mm) fuses. For operation at AC 100 to 120 V or AC 220 to 230 V, use 2A time lag fuses.

**⚠ WARNING**

**Risk of electrical shock.**

- ▶ The fuses should only be serviced after the AC power cord has been removed from the unit.

## 4 Incoming product acceptance and product 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. Comply with the permitted ambient conditions.

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

#### 4.1.1 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

#### 4.1.2 Manufacturer address

Endress+Hauser  
371 Parkland Plaza  
Ann Arbor, MI 48103  
USA  
[www.endress.com](http://www.endress.com)

### 4.2 Scope of delivery

The scope of delivery comprises:

- Calibration Accessory in the configuration ordered
- *Raman calibration accessory Operating Instructions*
- Calibration Accessory Certificate of Product Performance
- Local declarations of conformity, if applicable
- Certificates for hazardous zone use, if applicable
- Calibration Accessory optional accessories, if applicable

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

## 5 Operation

### 5.1 Calibration data file

Each Raman calibration accessory is supplied with a source spectral file on a memory stick. The file describes the relative spectral output of the accessory's halogen intensity lamp as measured outside the diffuser window. The halogen lamp has a nominal color temperature of 2840 K. However, the diffusers used in the lamp head modify the spectral output from that of a simple blackbody. Simple blackbody corrections are therefore strongly discouraged when using this accessory. The source spectral file is a more accurate characterization of the spectral output shape provided by the accessory when it is used with an optional adapter accessory.

It is important to distinguish between calibration of spectral output shape (color temperature) and absolute spectral output level ( $\text{watts/cm}^2/\text{sr/nm}$ ). This calibration accessory and the analyzer software that uses the calibration only correct the shape of measured spectra.

The resultant units of spectral intensity provided in the source spectral file are relative photon flux per wavenumber as a function of wavelength in nanometers. Although different in shape from traditional lamp units of  $\text{watts/nm}$  (by a factor of 3), these are the units preferred by Raman spectroscopists. CCD cameras used in Raman instruments register an output signal level in "counts," which is proportional to the photon flux at the detector and the quantum efficiency at the corresponding wavelength. Raman spectra are presented in terms of counts versus Raman shift, where Raman shift is specified in wavenumbers ( $\text{cm}^{-1}$ ). Therefore, the lamp head calibration is measured in terms of photons per wavenumber.

The software used to operate analyzers is configured to directly read the accessory's SSF in the instrument standardization process. The data provided in this file are specifically for use with analyzers and are not intended for general radiometric purposes.

### 5.2 Raman shift standard

A Raman shift standard is used in conjunction with the Raman calibration accessory. Depending on the application, Endress+Hauser recommends one of three Raman shift standards. One of these three shift standards is cyclohexane, which is used with the optical grade vial. Endress+Hauser recommends cyclohexane and provides a sealable optical-grade vial.

It is the user's responsibility to provide cyclohexane, CHROMASOLV, for HPLC,  $\geq 99.7\%$  (Sigma-Aldrich p/n 34855) and fill the supplied optical vial. The vial is mounted in a holder having similar mounting features as the lamp head. It accepts the same adapters for probe objectives.

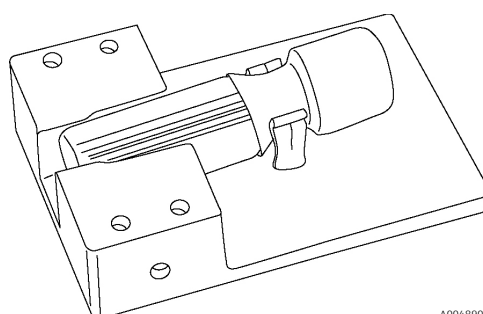


Figure 3: Cyclohexane Raman shift standard

#### NOTICE

- ▶ Contact your sales associate for specific questions related to your probe, optic, and sampling system.

Once the laser wavelength is known, the calibrated wavelength axis can then be accurately transformed into a calibrated Raman shift axis for accurate Raman measurement of unknown samples.

After intensity standardization with the tungsten-halogen source, the Raman shift standard performs final system verification over the operating range of the instrument. The analyzer verification success occurs when the measured cyclohexane peaks and amplitudes match established standards to within specified tolerances and as displayed in the verification report.

### 5.3 Raman calibration protocol

The protocol for use of the Raman calibration accessory differs among software packages. Instructions for use of both the wavelength and intensity source are provided in the associated analyzer operations manuals.

The recommended calibration and qualification sequence follows this order:

1. Calibrate the wavelength using the neon standard.
2. Calibrate or standardize the intensity using the tungsten-halogen standard.
3. Calibrate the laser wavelength using the Raman shift standard.
4. Qualify the calibration using the Raman shift standard.

Refer to the appropriate chapters in your analyzer's operating instructions for instructions for detailed instructions.

 **CAUTION**

- ▶ Give the lamp at least 12.5 minutes to warm up and stabilize before you perform the intensity standardization.
- ▶ The lamp turns off automatically after it has been on for 45 minutes. If the lamp has been on for more than 45 minutes and you have to set up an intensity sequence that lasts over 10 minutes, turn the halogen lamp off and back on to reset the 45-minute timer before beginning the process.
- ▶ For critical applications the position and focus are crucial. Position the probe as precisely as possible each time a calibration is performed. For best results this includes the rotation of the HCA adapter.

## 6 Maintenance

### 6.1 Lamp head and adapters

Neon and halogen light are both emitted from the diffuser window on the top surface of the lamp head. ¼-20 and 8-32 threaded holes are provided on the sides of the lamp head for convenient post mounting, if desired. Four 4-40 threaded holes surround the diffuser window for attachment of optional interface adapters.

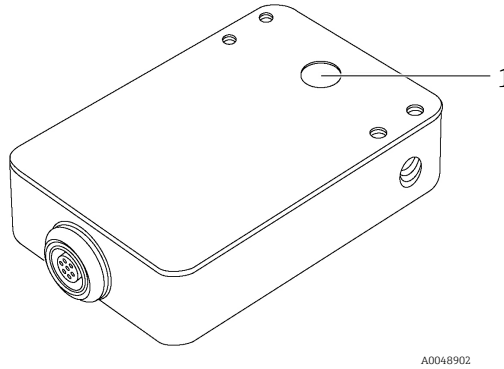


Figure 4: Lamp head - Diffuser Window (1)

The available interface adapters are listed in the table below.

Description	Part Number
FC fiber adapter	70187579
Non-contact objective adapter (NCO-0.4 and NCO-0.5 optics)	70188144
1.25 cm (0.5 in) Immersion optic (IO) adapter (also used for Rxn-40 and Rxn-41 probes)	70187580
6 mm optic	70193240 KOR20-AAAD
Rxn-20 probe adapter	70187749
0.635 cm (0.25 in) IO adapter	70187716
bIO immersion adapter	70187785

The lamp head attaches to an Endress+Hauser NCO-0.4 optic with the standard NCO-0.4/NCO-0.5 optics adapter. The immersion optic adapter is used with standard 1.25 cm (0.5 in) IOs, Rxn-40 probes, and Rxn-41 probes.

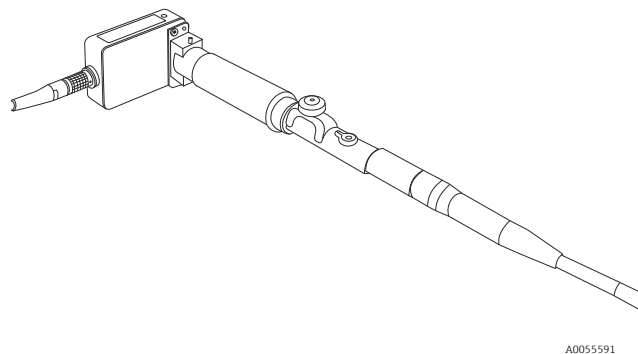
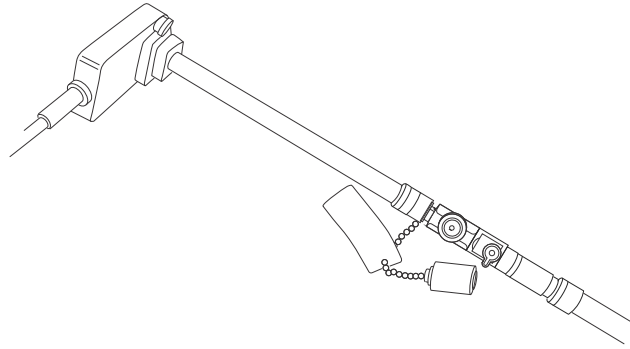


Figure 5: Lamp head on a 10x microscope objective



A0048617

*Figure 6: Lamp head on a 1.25 cm (0.5 in) immersion optic*

## 7 Repair

### 7.1 Servicing the unit and spare parts

The Raman calibration accessory is not user-serviceable and requires no routine maintenance. Endress+Hauser should perform all servicing and recertification of the Raman calibration accessory.

#### NOTICE

- ▶ Recertification of the intensity function is recommended once per year, or after 500 hours of halogen lamp ON time, whichever comes first.

Unit recertification consists of halogen lamp replacement (if needed), halogen lamp current adjustment (if needed), and spectral intensity output characterization. A new SSF is provided with the recertified unit. You must properly reference it in the Raman analyzer software in order for the recertification to be properly implemented.

#### NOTICE

- ▶ The performance of procedures (including service), the use of controls, or the adjusting of the instrument other than as specified in the manual will void the warranty.

## 8 Technical data

### 8.1 Specifications

Item	Description
Spectral intensity reference	Tungsten-halogen
Data file spectral range for given HCA models	HCA-532: 534.5 to 694.0 nm HCA-785: 790.7 to 1074.5 nm HCA-1000: 1012.6 to 1304.6 nm
Spectral intensity output repeatability (at time of certification)	< $\pm 0.65$ %
Spectral intensity output repeatability (over any 4000 $\text{cm}^{-1}$ spectrum, over 500 hours)	$\pm 2.65$ %
Total long term spectral uncertainty (at any wavelength)	HCA-532: $\pm 2.85$ % HCA-785: $\pm 6.05$ % HCA-1000: $\pm 10$ %
NIST traceable primary standard uncertainty	Available upon request
Power source	AC 100 to 240 V 50 Hz to 60 Hz
Power consumption	30 W maximum
Control unit dimensions	212 x 270 x 43 mm (8.3 x 10.6 x 1.7 in)
Control unit mass (weight)	1.60 kg (3.5 lb)
Lamp head dimensions	50 x 80 x 19 mm (2.0 x 3.1 x 0.7 in)
Lamp head mass (weight)	0.10 kg (0.2 lb)
CE certified	Yes



## 9 Supplementary documentation

All documentation is available:

- On the media device supplied (not included in the delivery for all device versions)
- On the Endress+Hauser mobile app: [www.endress.com/supporting-tools](http://www.endress.com/supporting-tools)
- In the Downloads area of the Endress+Hauser website: [www.endress.com/downloads](http://www.endress.com/downloads)

This document is an integral part of the document package, which includes:

Part Number	Document Type	Document Title
TI01637C	Technical Information	Raman calibration accessory technical information

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