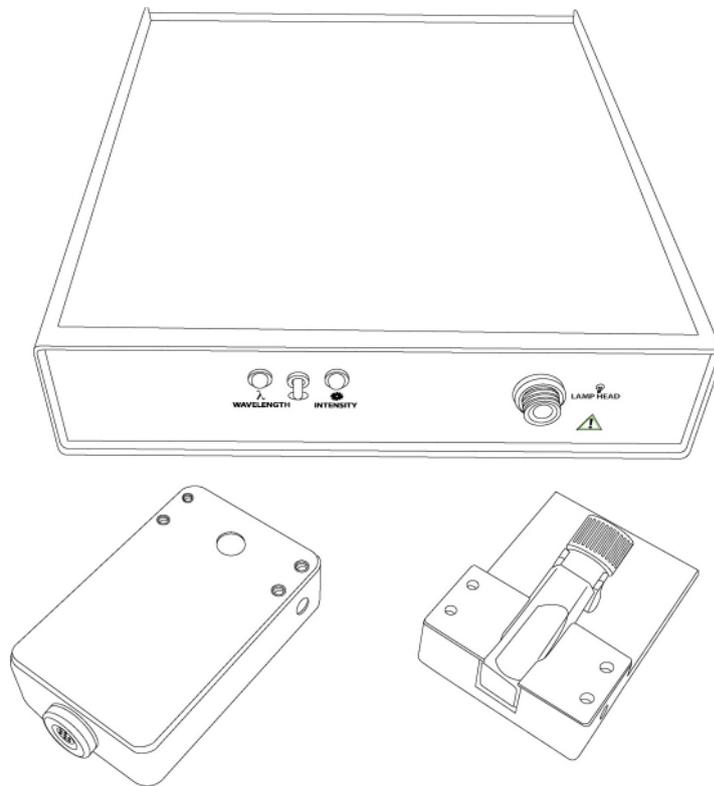


# Operating Instructions

## Raman Calibration Accessory





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# 1 About this document

## 1.1 Warnings

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

Table 1. Warnings

## 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).

Table 2. Symbols

## 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
°C	Celsius
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>

Table 3. Glossary

## 2 Basic safety instruction

### NOTICE

- ▶ The safety information in this section is specific to the Calibration Accessory. Refer to the *Raman Rxn2*, *Raman Rxn4*, and *Raman Rxn5 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 both wavelength and 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.

During operation:

1. If faults cannot be rectified: products must be taken out of service and protected against unintentional operation.

### 2.4 Product safety

The 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 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 Calibration Accessory.
- Do not look directly into the laser beam.
- Do not stare at diffused or reflection 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. These will 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 both wavelength and intensity. When used in conjunction with the calibration protocol recommended in this manual, it allows different instruments to be standardized such 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 wavelength and intensity reference lamps housed in a compact lamp head. The lamp head is connected to the control unit by a 6-foot (1.8 meter) 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.

Neon lamps in the lamp head provide a wide spectrum of atomic emission lines. These emission lines allow convenient wavelength calibration of a broad range of Raman analyzers that use different laser excitation wavelengths. The software provided with your analyzer is designed for use with this accessory and contains automated features for finding and identifying neon emission lines.

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.

Analyzer	Wavelength Axis	Intensity Axis	Laser Wavelength	Verification
Raman Rxn2	×	✓	×	✓
Raman Rxn4	×	✓	×	✓

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 those calibrations with those analyzer models.

Table 4. 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 a memory stick. This file is referred to as the Source Spectral File (SSF). Because the SSF is relative, not absolute, 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 conveniently reference the accessory's SSF in the instrument standardization process. Instrument intensity standardization corrects for instrument-to-instrument response variations.

These variations include the removal of:

- 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 the relevant performance of individual analyzers such that spectral, and hence chemical, models can be transferred to multiple analyzers.

Also provided with the Raman Calibration Accessory is an empty vial for the user to fill with cyclohexane for use as a Raman shift standard. This is used to calibrate the laser's operating wavelength. It is also used for subsequent qualification of the overall system calibration.

The lamp head and Raman shift standard vial are both sized to be conveniently 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, as well as Ferrule Connector (FC) and Subminiature Assembly (SMA) fiber connectors. For critical applications, it is recommended that the absolute position of the Raman Calibration Accessory (HCA) output relative to the probe head be tightly controlled.

Endress+Hauser recommends unit recertification and halogen lamp replacement after 1 year or 500 hours of burn time respectively. To maximize the life of the accessory, the halogen lamp will automatically turn off after 60 minutes of uninterrupted “on” time. You may, of course, turn the lamp off manually at any time. The control unit also

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 section 3.1.3.

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.

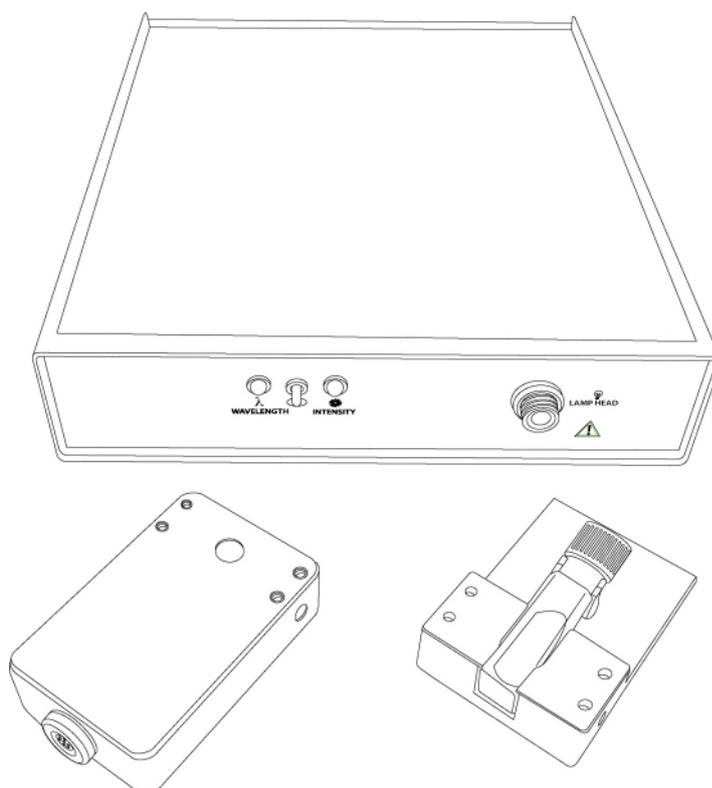


Figure 1. Front view of the control unit with the lamp head

The figure below is a rear view of the control unit, showing the power connection, the unit power ON/OFF switch, the fuse door and a Universal Serial Bus (USB) communications port.

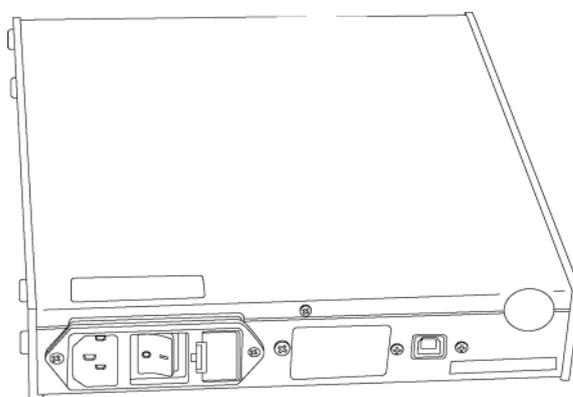


Figure 2. Rear view of the control unit

#### NOTICE

- The USB port is for factory use only. The communication protocol is proprietary and the user should not attempt communication since damage could occur.

### 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 6-foot (1.8-meter) cable connecting the lamp head and the control unit has opposing connector genders 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

Pressing 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 60 minutes. The base unit keeps track of the lamp's elapsed "on" time (to the nearest 0.1 minute). If the lamp time exceeds 450 hours, the LED indicator will be continuously yellow when lit. If lamp time exceeds 500 hours, the LED indicator will be continuously red when lit. These two indications alert you to return the unit to Endress+Hauser for recertification.

It is recommended that the Halogen lamp be allowed to warm up for 12.5 minutes for the color temperature of the bulb to fully stabilize prior to its use. The Halogen lamp LED indicator will continue blinking as a visual indicator of bulb warm-up time and will be solid when the warm-up period is complete.

The halogen lamp can be turned off by pressing the front panel switch to the right again or pressing it to the left to light the neon lamps.

### 3.1.4 Neon lamps on/off

The neon lamps can be lit by pressing the front panel switch to the left. There is no time-out function.

The neon lamps are extinguished by pressing the switch to the left again or pressing it to the right to light the halogen lamp.

### 3.1.5 Power

The Raman Calibration Accessory uses a universal input switching power supply and will operate over a line input range of 100–240 VAC 50-60 Hz. Main (line) power connection is made through a standard power cord with universal IEC320 connector. Power consumption is 30 Watt (W) maximum.

### 3.1.6 Fuses

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

#### WARNING

- ▶ The fuses should only be serviced after the AC power cord has been removed from the unit to avoid electrical shock situations.

## **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. Make sure to comply with the permitted ambient conditions.

If you have any questions, please 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

### **4.2 Scope of delivery**

The scope of delivery comprises:

- Calibration Accessory in the configuration ordered
- 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: Please 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 (halogen intensity lamp) as measured outside the diffuser window. The halogen lamp inside the lamp head has a nominal color temperature of 2840 K. However, the diffusers used in the lamp head significantly 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 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 use it are used only to 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 very different in shape from the more 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 normally presented in terms of counts versus Raman shift, where Raman shift is specified in wavenumbers ( $\text{cm}^{-1}$ ). Therefore, the lamp head calibration is 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 provide 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 required for use of the Raman Calibration Accessory. Endress+Hauser recommends cyclohexane and provides a sealable optical-grade vial.

#### **CAUTION**

- ▶ 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 and fiber connectors.

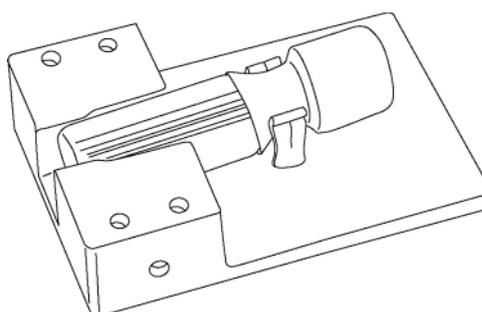


Figure 3. Cyclohexane Raman shift standard

#### **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.

The Raman shift standard may also be used to perform final calibration verification over the entire operating range of the instrument after intensity standardization with the tungsten halogen source. The analyzer calibration is verified when the measured cyclohexane peak positions and amplitudes match the established standards to within specified tolerances.

### 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.

A summary of the recommended calibration/qualification sequence is as follows:

1. Wavelength calibration (neon standard).
2. Intensity calibration/standardization (tungsten-halogen standard).
3. Laser wavelength calibration (Raman shift standard).
4. Calibration qualification (Raman shift standard).

#### **⚠ CAUTION**

- ▶ The halogen lamp should be allowed at least 12.5 minutes to warm up and stabilize before you perform the intensity standardization. Also, the lamp turns off automatically after it has been on for 60 minutes. If the lamp has been on for more than 45 minutes and you have set up a very long intensity sequence (that may last for over 10 minutes), you should turn the halogen lamp off and back on to reset the 60-minute timer before beginning the process.
- ▶ For critical applications the position and focus are crucial. Care should be taken to position the probe as precisely as possible each and every 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. 1/4-20 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.

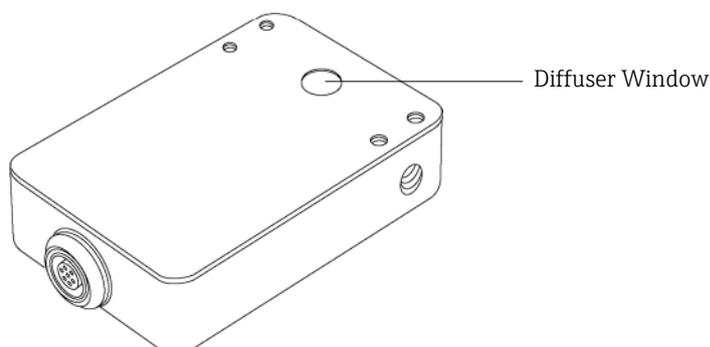


Figure 4. Lamp head

The available interface adapters are listed in the table below.

Description	Model Number	Part Number
FC fiber adapter	HCA-FC	2004645-101
SMA fiber adapter	HCA-SMA	2004649-101
Non-contact objective adapter (NCO-0.4 and NCO-0.5 optics)	HCA-MO-NIR-10X	2004651-101
Non-contact 1.3 inch adapter (NCO-1.3)	HCA-NCO-1.3	2005243-101
Non-contact 2.5 inch adapter (NCO-2.5)	HCA-NCO-2.5/3	2005244-101
½ inch Immersion Optic (IO) adapter (also used for Rxn-40 and Rxn-41 probes)	HCA-IO	2004647-101
Rxn-20 probe adapter	HCA-RXN-20	2008556-101
Quarter-inch IO adapter	HCA-IO-0.25	2006980-101
bIO immersion adapter	HCA-bIO	2012724

Table 5. Available interface adapters

The lamp head attached 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 ½ inch IOs, Rxn-40 probes and Rxn-41 probes.

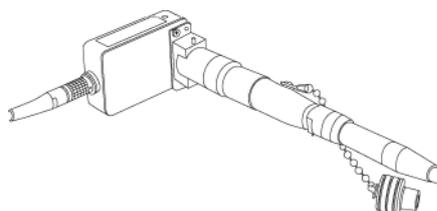


Figure 5. Lamp head on a 10x microscope objective

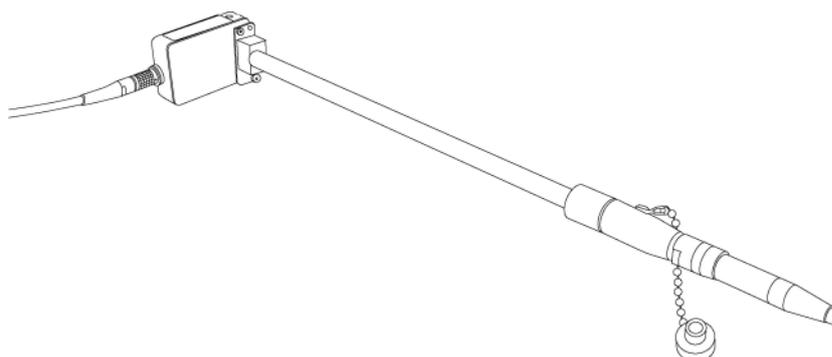


Figure 6. Lamp head on a half-inch immersion optic

## 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.

The wavelength calibration function of the unit never needs recalibration. Atomic emission lines from the neon lamps do not change over time. In the unlikely event that the neon lamps fail during the life of the unit, the lamp head can be returned to Endress+Hauser for service.

#### 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.

The following table lists parts that can be ordered and installed.

Part Number	Description
6060018	2A fuse (for 100–120 VAC or 220–240 VAC, 2 required).
6060003	Power cord (U.S. style).

Table 6. Replacement parts

## 8 Technical data

### 8.1 Specifications

Item	Description
Wavelength reference	Neon lamps (2)
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	100–240 VAC 50–60 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 lbs)
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 lbs)
CE certified	Yes

Table 7. Specifications

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