

Operating Instructions

Raman flow assembly calibration and verification kit



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1 About this document

1.1 Warnings

Structure of Information	Meaning
⚠ WARNING Causes (/consequences) 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.
⚠ 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 Symbols on the device

Symbol	Description
	The Laser Radiation symbol is used to alert the user to the danger of exposure to hazardous visible and invisible laser radiation when using the system.
	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.

Table 2. Symbols

1.3 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.

The Export Control Classification Number (ECCN) of the Raman flow assembly calibration and verification kit is EAR99.

1.4 Glossary

Term	Description
°C	Celsius
CCD	charge coupled device
cm	centimeter
CRS	calibration reference standard
°F	Fahrenheit
in	inches
IP	ingress protection
IPA	isopropyl alcohol
kg	kilogram
lbs	pounds
mm	millimeter
nm	nanometer
SSF	source spectral file
WEEE	Waste Electrical and Electronic Equipment

Table 3. Glossary

2 Basic safety instruction

The safety information in this section is specific to the Raman flow assembly calibration and verification kit that is compatible with Raman spectroscopic probes. Refer to the applicable probe operating instructions for additional information related to probe and laser safety.

2.1 Requirements for personnel

- Installation, commissioning, operation, and maintenance of the Raman flow assembly calibration and verification kit 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 corrected by authorized trained personnel. Repairs not described in this document must be carried out only directly at the manufacturer's site or by the service organization.

2.2 Designated use

The Raman flow assembly calibration and verification kit is used for standardizing Raman instruments and analyzers to give precise spectral intensity measurements.

2.3 Workplace safety

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

- Installation guidelines
- Local standards and regulations electromagnetic compatibility

2.4 Operational safety

Before commissioning the entire measuring point:

- Verify that all connections are correct.
- Ensure that electrical cables and optical fiber connections are undamaged.
- 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.

CAUTION

Activities while the micro flow bench calibration or verification cell is in operation introduce risk of exposure to measured materials.

- ▶ Follow standard procedures for limiting exposure to chemical or biological materials.
- ▶ Clean any spills using the appropriate site policies and cleaning procedures.

2.5 Important safeguards

- Do not use the micro flow bench calibration or verification cells for anything other than their intended use.
- Do not look directly into the laser beam.
- Do not point a laser at a mirrored surface or introduce a mirrored object into the micro flow bench beam-path.
- Do not leave attached and unused probes uncapped or unblocked.
- If an emission shutter is available on the probe (e.g., Rxn-10 probe), ensure the shutter is in the closed position when installing or removing the calibration or verification cell from the micro flow bench.
- Always use a laser beam block.

2.6 Product safety

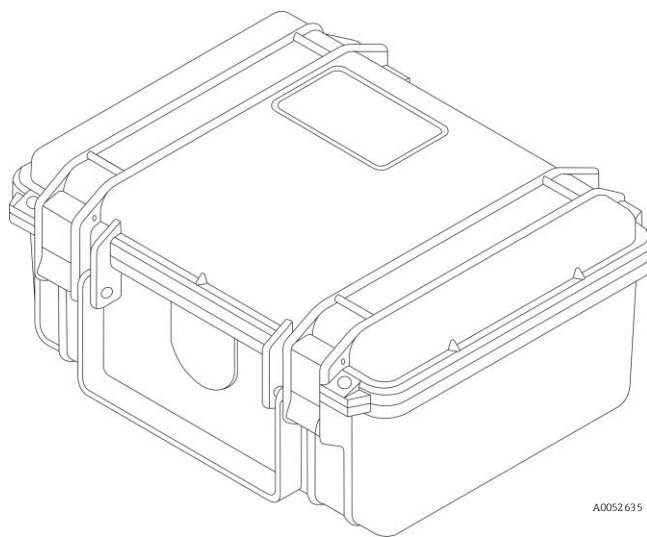
The Raman flow assembly calibration and verification kit has been designed to meet state-of-the-art safety requirements, has been tested, and left the factory in a condition in which it is safe to operate. The relevant regulations and international standards have been observed. Devices connected to Raman analyzers must comply with the applicable safety standards.

3 Product description

The Raman flow assembly calibration and verification kit contains all the necessary hardware for calibrating and verifying the calibration of the micro flow bench. The contents include:

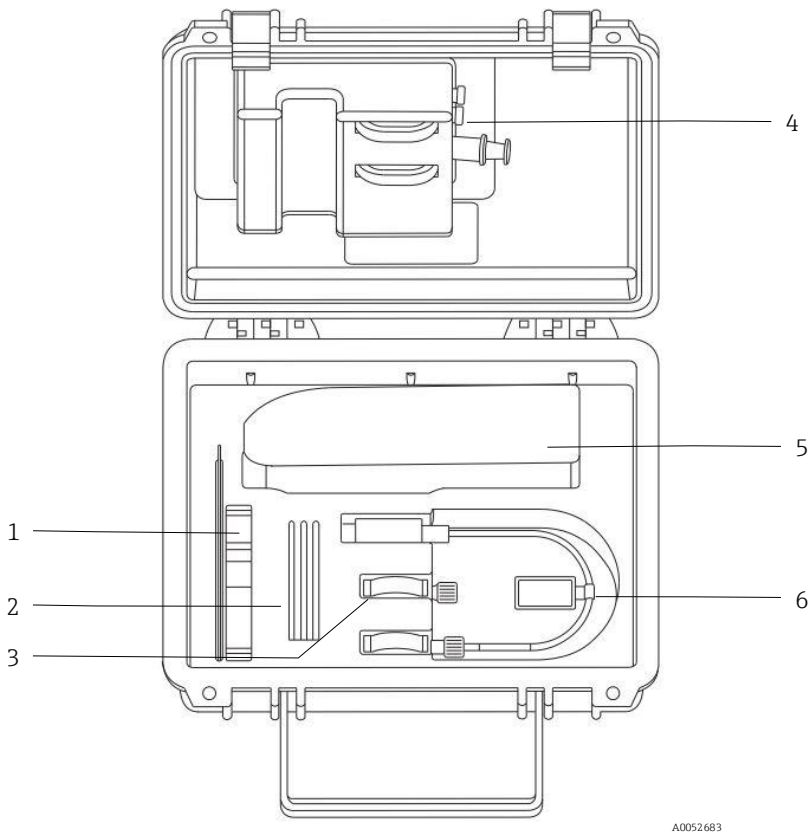
- Micro flow bench calibration cell
- Micro flow bench verification cell
- Temperature display
- Flash drive with calibration files
- Syringe
- Syringe tips (2)
- Lens cleaning wipes (3)

The external and internal views of the Raman flow assembly calibration and verification kit are shown below.



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Figure 1. External view of the Raman flow assembly calibration and verification kit



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Figure 2. Internal view of the Raman flow assembly calibration and verification kit

#	Description
1	Flash drive
2	Lens wipes
3	Micro flow bench verification cell
4	Syringe and tips
5	Temperature display
6	Micro flow bench calibration cell and temperature sensor connector

3.1 Micro flow bench calibration cell

The micro flow bench calibration cell is used for standardizing Raman instruments and analyzers to give precise spectral intensity measurements. When used with the calibration protocol in this manual, the calibration cell ensures different instruments generate similar spectra when measuring a given sample. The micro flow bench calibration cell was created specifically for use with Raman instruments and analyzers manufactured by Endress+Hauser.

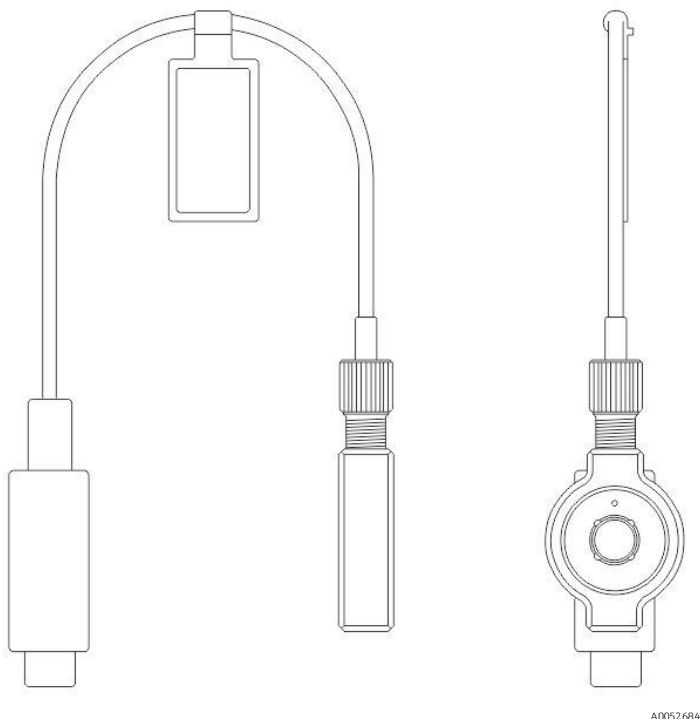


Figure 3. Micro flow bench calibration cell

All Endress+Hauser Raman analyzers have built-in wavelength and laser wavelength calibration. In addition, the micro flow bench calibration cell includes signal from the analyzer's specific fiber, probe, and optic to standardize the integrated system. This reduces variation between other instruments by producing a standardized spectral response.

For intensity standardization, a calibration reference standard (CRS) provides a factory-characterized spectral output. A wavelength-specific CRS is required for each Raman excitation wavelength.

The standard micro flow bench calibration cell interfaces with the Endress+Hauser micro flow bench and should be configured in the same manner as the micro flow bench. Please consult the manufacturer for non-standard configurations.

3.2 Micro flow bench verification cell

The micro flow bench verification cell is used to confirm that the calibration process has resulted in standardizing the instrument back to factory specifications. This is accomplished by collecting a Raman spectrum of the reference sample contained within the verification cell and comparing it to preset criteria for the expected spectral response. If the analyzer passes verification, the instrument is properly standardized. If the analyzer fails verification, this could indicate recalibration of the analyzer and probe is needed. Regular performance of the verification step is recommended to ensure the instrument is properly calibrated and standardized.

The micro flow bench verification cell was created specifically for use with Raman instruments and analyzers manufactured by Endress+Hauser.

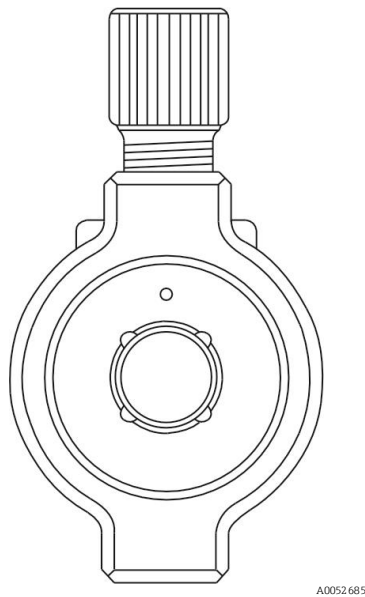


Figure 4. Micro flow bench verification cell

The standard sample initially provided and required for use with the micro flow bench verification cell is 70 % isopropyl alcohol (IPA).

NOTICE

Only 70 % IPA should be used for optical verification.

- ▶ Only 70 percent by volume (%v/v) will work. Endress+Hauser recommends using CiDehol 70 by Decon Laboratories.
- ▶ Use of any other liquid for verification will result in a failed verification and may result in damage to both the verification accessory and the Raman probe.

3.3 Temperature display

A temperature display with the recommended precision and accuracy is provided with the calibration kit and connects to the temperature measurement sensor contained within the micro flow bench calibration cell.

To ensure proper results during probe calibration, the micro flow bench calibration cell temperature must be entered into the Raman RunTime software when prompted. This requires a Raman analyzer with Raman RunTime 6.2.2+ embedded software.

Failure to enter an acceptable temperature value may result in an improperly calibrated probe. Refer to the Raman RunTime software manual for instructions to input the temperature value.

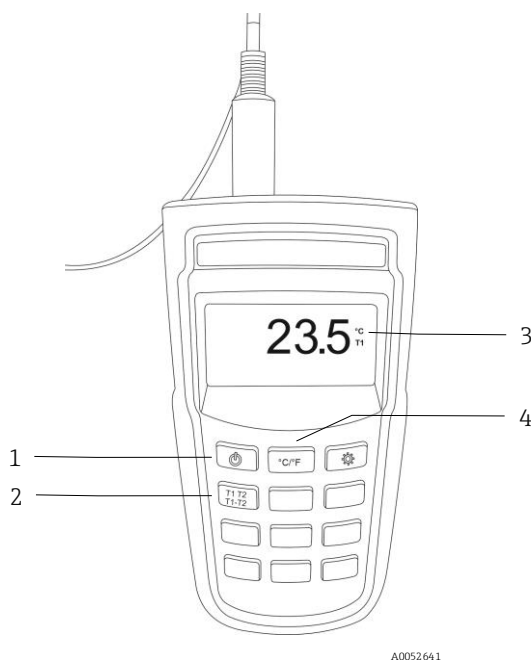


Figure 5. Temperature display

#	Description
1	Power button
2	Probe input selection (T1 probe, T2 probe, or difference between the two probes)
3	Main display showing temperature reading, temperature units, and selected probe input
4	Temperature unit selection (°C or °F)

3.4 Flash drive with calibration files

A software file detailing the spectral characteristics of the device, as well as an electronic certificate, is provided with each unit on a flash drive when new and when recertified.

A spectral data file (.spc format) containing the spectral output of the calibration cell is provided on the flash drive and is also stored internally on the hard drive of the analyzer after the first time the probe is calibrated.

The software provided with the analyzer requires the calibration accessory source spectral file (SSF) as part of the instrument standardization process. Instrument intensity standardization corrects for instrument-to-instrument response variations. Ensure that the serial number for the micro flow bench calibration cell used matches the serial number for the SSF on the flash drive or hard drive any time a probe calibration is executed. Because the SSF is relative, use of this accessory corrects only the normalized shape of measured spectra, not the absolute magnitude.

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

3.5 Syringe and tips

A syringe with leur lock tips (2) is included in the kit for servicing the micro flow bench verification cell. Refer to Replacing the verification sample →  for maintenance instructions.

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.

4.2 Product identification

4.2.1 Label

At a minimum, the Raman flow assembly calibration and verification kit is labeled with the following information:

- Endress+Hauser branding
- Extended order code
- Product description

The micro flow bench calibration and verification cells are labeled with the following information:

- Serial number
- Part number
- Calibration sticker (calibration cell only)

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:

- Raman flow assembly calibration and verification kit in the configuration ordered
- *Raman flow assembly calibration and verification kit Operating Instructions* manual
- Raman flow assembly calibration and verification kit Certificate of Product Performance
- Local declarations of conformity, if applicable
- Certificates for hazardous zone use, if applicable
- Raman flow assembly calibration and verification kit optional accessories, if applicable

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

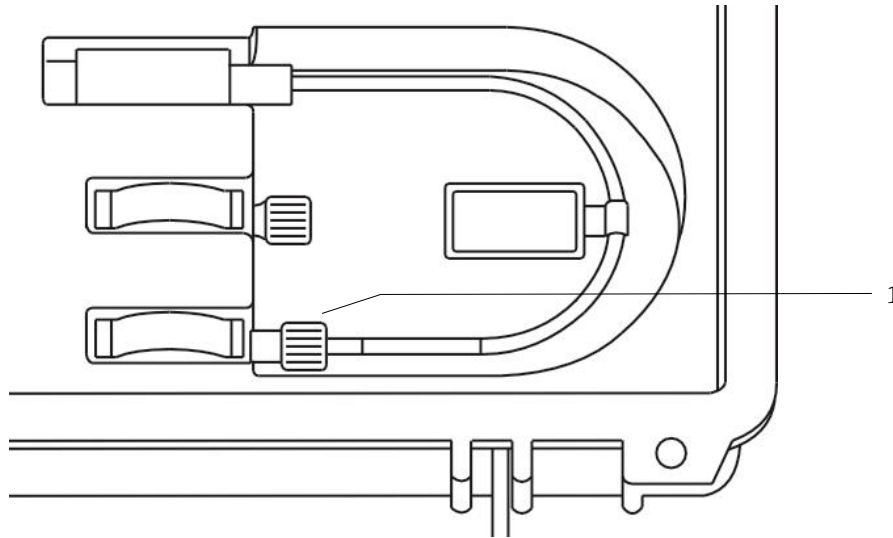
5 Installation

The micro flow bench calibration and verification cells are designed to install into the micro flow bench in the same manner as a standard micro flow cell. Refer to the *Raman flow assembly Operating Instructions* for detailed installation instructions.

5.1 Micro flow bench calibration cell

To install the micro flow bench calibration cell into the micro flow bench:

1. Ensure the probe, micro flow bench and calibration cell are at ambient temperature.
 - See Technical data →  for more information.
 - Failure to operate at ambient temperature can cause incorrect calibrations.
2. Using clean gloves, remove the micro flow bench calibration cell from the case.
 - Handle by the plastic nut, taking care not to touch the window surfaces or excessively strain the temperature-sensor leads.
 - Clean gloves are recommended whenever handling the micro flow bench calibration cell to minimize the transmission of oils or other film-type contamination to the exterior optical window surfaces.



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Figure 6. Micro flow bench calibration cell with location of plastic nut (1) for proper handling and removal from the case

3. Perform a visual inspection to ensure the optical surfaces of the micro flow bench calibration cell are clean prior to calibration.
 - Use of an illuminated stereo microscope configuration with a minimum 10x magnification is recommended.
 - Both normal and off-axis inspections are recommended to check for particle and film-type contamination.
 - If cleaning is required, refer to Cleaning the optical surfaces →  for instructions.

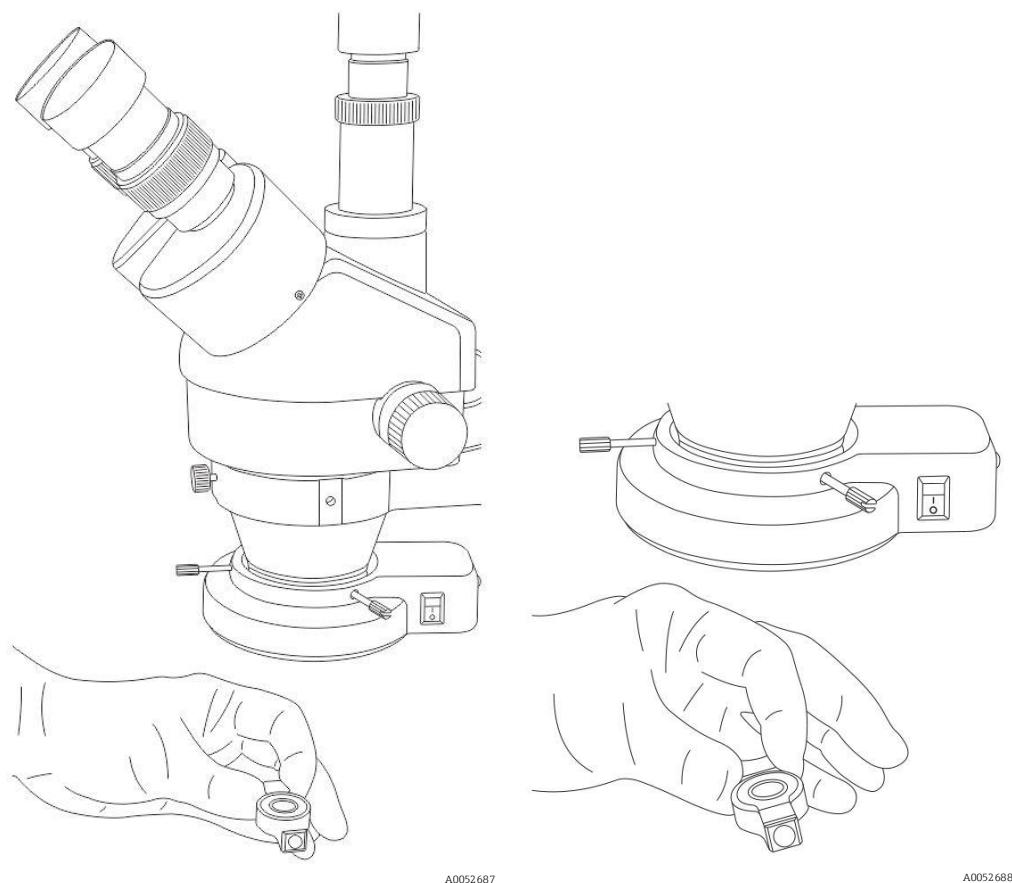


Figure 7. Normal (left) and off-axis (right) inspection of a cell under a microscope

4. To install the micro flow bench calibration cell into the micro flow bench, retract the light shield and align the calibration cell with the probe markings toward the source.

It is recommended that the beam dump is open to maximize precision while calibrating the optical system. Prior to calibration, ensure that the beam dump is in the correct position.

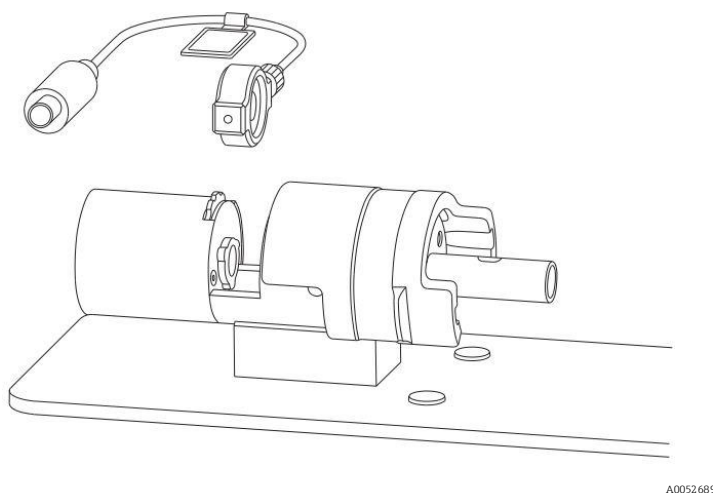
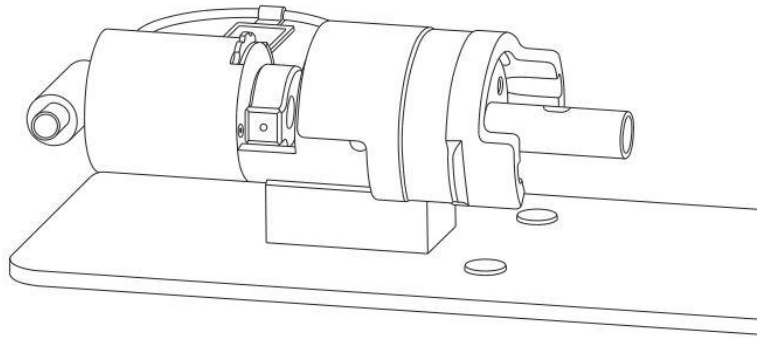


Figure 8. Retracting the micro flow bench light shield and aligning the calibration cell

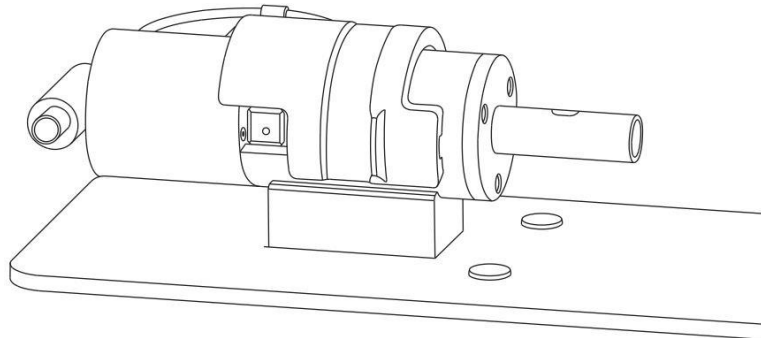
5. Once the micro flow bench calibration cell is in position, move the light shield forward until the plunger contacts the cell and holds it in place.



A0052.690

Figure 9. Micro flow bench calibration cell in position

6. Close the micro flow bench light shield completely over the calibration cell.



A0052.691

Figure 10. Closing the micro flow bench light shield

7. If the calibration cell is no longer at ambient temperature (e.g., because of handling during inspection and cleaning), allow the cell to return to ambient temperature and stabilize for at least 5 minutes before calibrating. Refer to Measure the micro flow bench calibration cell temperature →  for instructions.

⚠ WARNING

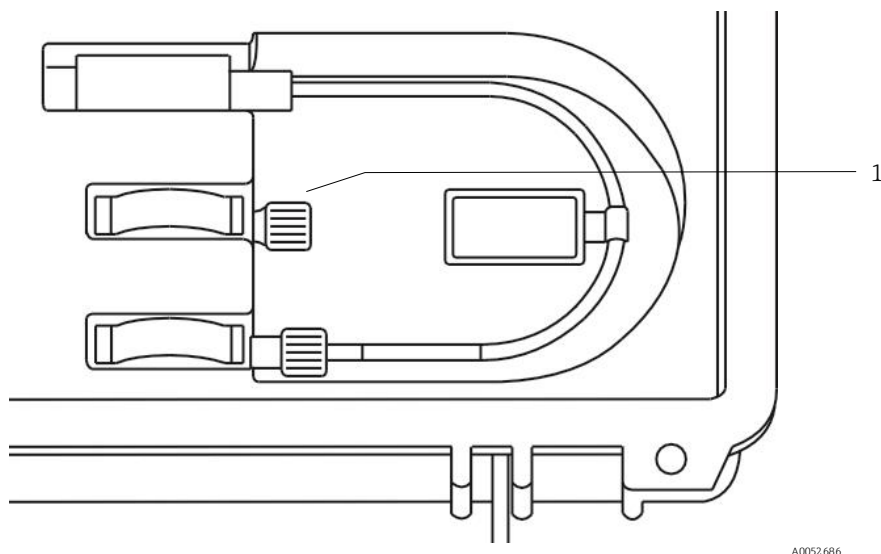
Do not remove the micro flow bench calibration cell from the micro flow bench during calibration or while the laser is on.

- Serious injury can result from exposure to the laser.

5.2 Micro flow bench verification cell

To install the micro flow bench verification cell into to micro flow bench:

1. Ensure the probe, micro flow bench and verification cell are at ambient temperature.
 - See Technical data →  for more information.
 - Failure to operate at ambient temperature can cause incorrect verifications.
2. Using clean gloves, remove the micro flow bench verification cell from the case.
 - Handle by the plastic nut, taking care not to touch the window surfaces.
 - Clean gloves are recommended whenever handling the micro flow bench verification cell to minimize the transmission of oils or other film-type contamination to the exterior optical window surfaces.

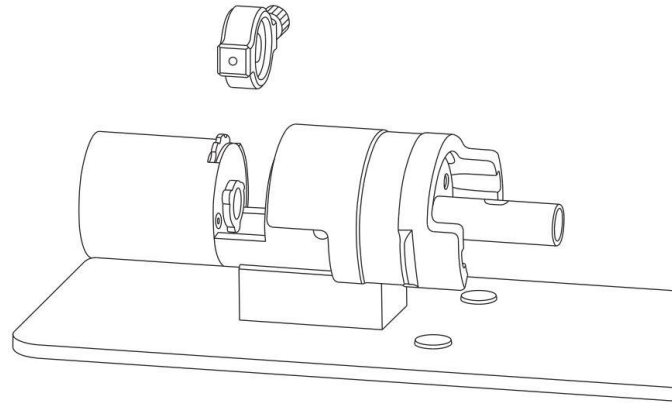


A0052.686

Figure 11. Micro flow bench verification cell with location of plastic nut (1) for proper handling and removal from the case

3. Perform a visual inspection to ensure the optical surfaces of the micro flow bench verification cell are clean prior to calibration.
 - Use of an illuminated stereo microscope configuration with a minimum 10x magnification is recommended.
 - Both normal and off-axis inspection are recommended to check for particle and film-type contamination.
 - If cleaning is required, refer to Cleaning the optical surfaces →  for instructions.
4. To install the micro flow bench verification cell into the micro flow bench, retract the light shield and align the verification cell with the probe markings toward the source.

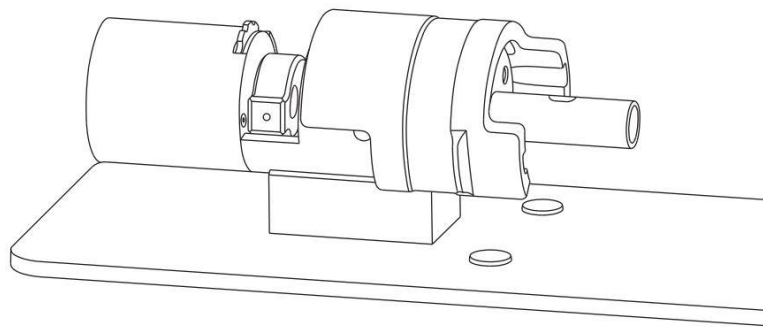
It is recommended that the beam dump is open to maximize precision while verifying the optical system. Prior to verification, ensure that the beam dump is in the correct position.



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Figure 12. Retracting the micro flow bench light shield and aligning the verification cell

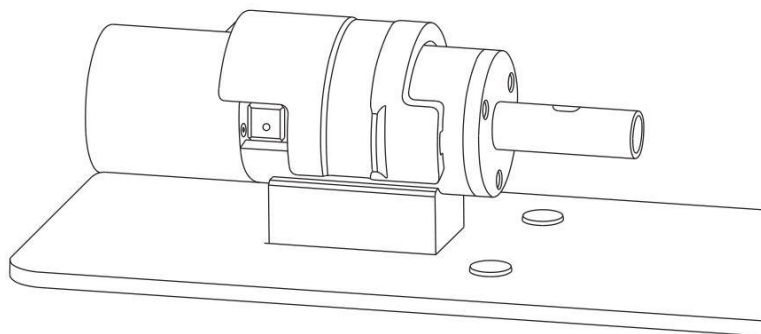
5. Once the micro flow bench verification cell is in position, move the light shield forward until the plunger contacts the cell and holds it in place.



A0052693

Figure 13. Micro flow bench verification cell in position

6. Close the micro flow bench light shield completely over the verification cell.



A0052694

Figure 14. Closing the micro flow bench light shield

⚠ WARNING

Do not remove the micro flow bench verification cell from the micro flow bench during verification or while the laser is on.

- Serious injury can result from exposure to the laser.

6 Operation

6.1 Measure the micro flow bench calibration cell temperature

During the calibration process, the micro flow bench calibration cell temperature is measured and entered into the Raman RunTime 6.2.2+ embedded software on the Raman analyzer.

To obtain the micro flow bench calibration cell temperature:

1. Attach the temperature sensor connector to the temperature display port marked channel 1.
2. Turn on the temperature display using the red power button.
3. Verify the display reads T1 to the right of the main temperature reading. Toggle to T1 if needed.
4. Read the temperature shown on the display and enter it into the Raman RunTime software on the analyzer.
 - Make sure the temperature units selected on the display (°C or °F) match the units selected in Raman RunTime.
 - Refer to the Raman RunTime software manual for instructions to input the temperature value.



Figure 15. Thermometer inserted into the temperature measurement port

6.2 Accessory calibration data file

Each micro flow bench calibration cell is supplied with a source spectral file on a flash drive. The file describes the relative spectral output of the device as measured through the micro flow bench system. The software used to operate analyzers is configured to directly read the device'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.

6.3 Raman calibration and verification protocol

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

- Perform internal analyzer calibration. This may include alignment calibration, full wavelength calibration, or full laser wavelength calibration depending on the status of the analyzer.
- Perform probe calibration using the micro flow bench calibration cell in the micro flow bench as a system.
- Perform probe verification using the micro flow bench verification cell in the micro flow bench as a system to verify the calibration results.
- View calibration and verification reports.

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 is highly recommended.

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

6.4 Removing the calibration and verification cells

The micro flow bench calibration and verification cells are designed to be removed from the micro flow bench in the same manner as a standard micro flow cell. Refer to the *Raman flow assembly Operating Instructions* for detailed removal instructions.

NOTICE

- Before using the calibration or verification cells with an additional micro flow bench, perform a visual inspection of the optical surfaces and clean as needed.

6.5 Storing the micro flow bench calibration cell

When stored or not in use, the micro flow bench calibration cell should be safely inserted into the case-insert and the temperature sensor leads coiled and packed as shown on the cover of this manual. This protects the cell from damage and inadvertant contamination.

6.6 Storing the micro flow bench verification cell

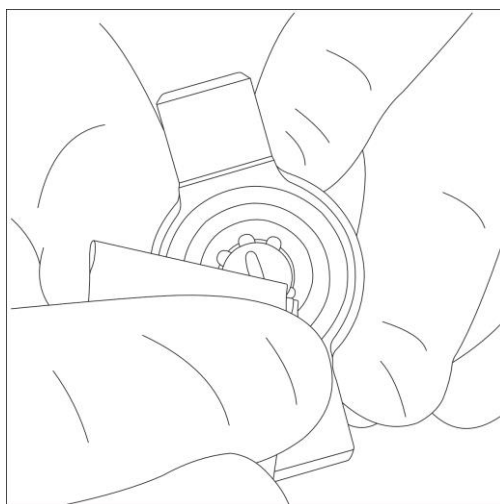
When stored or not in use, the micro flow bench verification cell should be safely inserted into the case-insert as shown in Figure 11. This protects the cell from damage and inadvertant contamination.

7 Maintenance

7.1 Cleaning the optical surfaces

The optical surfaces of the micro flow bench calibration cell and verification cell must be clean prior to use. If cleaning is required, Endress+Hauser recommends the process below. Clean gloves are recommended whenever handling the micro flow bench calibration or verification cell to minimize the transmission of oils or other film-type contamination to the exterior optical window surfaces.

Contaminant	Cleaning process
Large particles and dust	- Blow off the surface with clean, dry air. NOTICE Ensure only clean air is used. - Oil contamination in the air lines can result in film on the optical surface. - Inspect the surface. Repeat the cleaning process if large particles or dust remain.
Small particles	- Gently clean with the lens wipes provided in the Raman flow assembly calibration and verification kit. - Do not damage the optical surface with excessive or aggressive cleaning. - Use a fresh cleaning wipe to avoid scratching the surface with trapped debris. - Minimize liquid buildup on the window surface to avoid leaving film contamination on the surface. - Inspect the surface. Repeat the cleaning process if contaminants remain.
Film-type contamination	- Gently clean with the lens wipes provided in the Raman flow assembly calibration and verification kit. - Do not damage the optical surface with excessive or aggressive cleaning. - Use a fresh cleaning wipe to avoid scratching the surface with trapped debris. - Minimize liquid buildup on the window surface to avoid leaving film contamination on the surface. - Inspect the surface. Repeat the cleaning process if contaminants remain.



A0052695

Figure 16. Cleaning the optical surface with a lens wipe

7.2 Replacing the verification sample

The micro flow bench verification cell houses the verification sample. The standard sample initially provided and required for use with the accessory is 70 % isopropyl alcohol (IPA).

NOTICE

Only 70 % IPA should be used for optical verification.

- ▶ Only 70 percent by volume (%v/v) will work. Endress+Hauser recommends using CiDehol 70 by Decon Laboratories.
- ▶ Use of any other liquid for verification will result in a failed verification and may result in damage to both the verification accessory and the Raman probe.

It is recommended that the verification sample is replaced when:

- there is noticeable sample evaporation
- air bubbles are present
- probe verification fails

To replace the verification sample:

1. With clean gloves, hold the outer cell housing and unscrew the fill cap by turning it counter-clockwise. Set the cap aside.

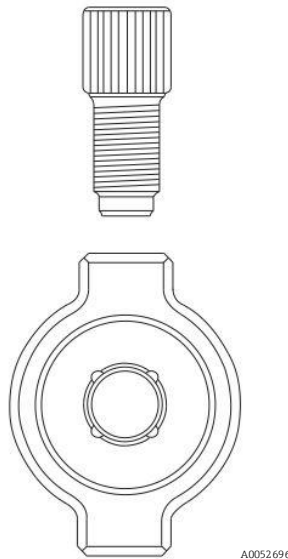
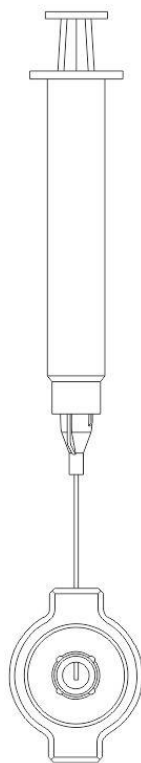


Figure 17. Micro flow bench verification cell with fill cap loosened

2. Remove the syringe and leur lock tip from the kit lid storage location, and install the leur lock tip onto the syringe.
3. Extract the existing verification sample from the verification cell.
 - Feed the leur lock tip into the fill port hole (where the fill cap was removed).
 - Extend the needle to the bottom of the vial. This can be viewed when looking into the containment window.
 - Extract the sample and discard it.



A0052697

Figure 18. Needle inserted into verification cell

4. Add the new verification sample to the verification cell.
 - Draw 70 % IPA into the syringe and discard. Repeat a few times to flush potential contaminants out of the syringe and tip.
 - Draw approximately 1 mL of 70 % IPA verification sample into the syringe.
 - Hold the syringe vertically with the tip pointing up, and expel all air bubbles.
 - Hold the micro flow bench verification cell with the fill port facing up to prevent any bubbles from getting trapped while filling.
 - Feed the leur lock tip down into the fill port hole, and inject the fresh verification sample into the sample space.
 - During filling, push any bubbles out of the dead volume. A small bubble will not impede the verification results, but a large bubble within the interrogation volume may skew verification results.
5. Replace the fill cap and turn clockwise until finger tight. Do not strip the threads on the cap.
6. Inspect the verification cell to ensure the optical surfaces are clean. Refer to Micro flow bench verification cell →  (step 3) for inspection instructions. If cleaning is required, refer to Cleaning the optical surfaces →  for instructions.

7.3 Recertifying the micro flow bench calibration cell

Unless noted differently for a specific application, Endress+Hauser recommends recertification of the micro flow bench calibration cell after 12 months to ensure that degradation to moisture has not occurred.

A new SSF is provided with the recertified calibration cell. Reference the new SSF when calibrating the system from that point forward.

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

8 Repair

8.1 Repairing the Raman flow assembly calibration and verification kit

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.

8.2 Replacement parts

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

9 Technical data

The specifications for the Raman flow assembly calibration and verification kit are listed below.

Item	Description
Spectral intensity reference	calibration reference standard (CRS)
Data file spectral range	CRS-785: 790.7 to 1074.5 nm
Spectral intensity output	< ± 2 %
Total long term spectral uncertainty (at any wavelength)	CRS-785: ± 6.05 %
Dimensions	kit: 235 x 192 x 85 mm (9.3 x 7.6 x 3.4 in) calibration cell: 24.5 x 10.0 x 47.7 mm (1.0 x 0.4 x 1.9 in) verification cell: 24.5 x 10.0 x 44.6 mm (1.0 x 0.4 x 1.8 in)
Calibration kit weight	1.5 kg (3.3 lbs)
IP rating	IP20
Operating conditions	0 to 40 °C (32 to 104 °F) < 80 % humidity, non-condensing
Recommended storage conditions	-15 to 50 °C (5 to 122 °F) < 80 % humidity, non-condensing

Table 4. Specifications

10 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
TI01720C	Technical Information	Raman flow assembly calibration and verification kit Technical Information

Table 5. Supplementary documentation

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