

Operating Instructions

Raman Calibration Tool

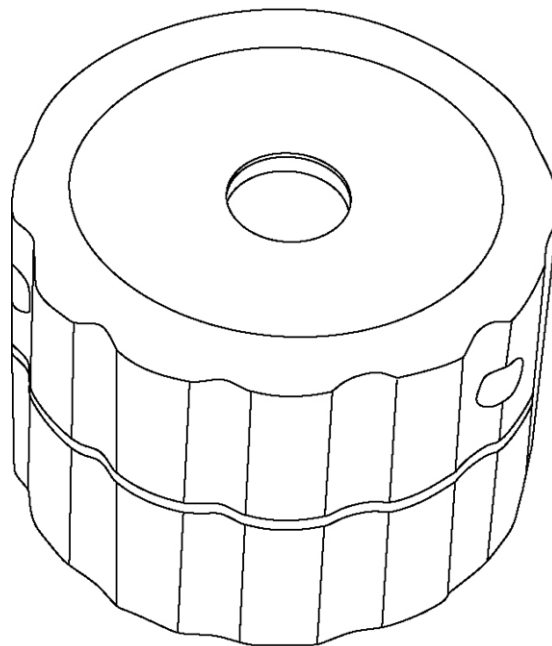


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1 About this document

1.1 Warnings

Structure of Information	Meaning
⚠ WARNING Causes (/consequences) If necessary, 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) If necessary, 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 If necessary, 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 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
EEA	European Economic Area
GRAMS	GRAMS AI program from Thermo Scientific
kg	Kilogram
lbs	Pounds
mm	Millimeter
NIST	National Institute of Standards and Technology
nm	Nanometer
RCT	Raman Calibration Tool
SRM	NIST Standard Reference Material
SSF	Source Spectral File
W	Watt
WEEE	Waste Electrical and Electronic Equipment

Table 3. Glossary

2 Basic safety instruction

NOTICE

- ▶ The safety information in this section is specific to the Calibration Tool. Refer to the *Raman Rxn2*, *Raman Rxn4*, and *Raman Rxn5 Operating Instructions* for additional analyzer-related safety information about working with lasers.

2.1 Requirements for the personnel

- Installation, commissioning, operation, and maintenance of the Calibration Tool may be carried out only by specially trained technical personnel.
- The technical personnel must be authorized by the plant operator to carry out the specified activities.
- Any electrical connections must be performed by trained or certified electrical technician.
- The technical personnel must have read and understood these Operating Instructions and must follow the instructions contained therein.
- Faults at the measuring point may only be rectified 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 Calibration Tool is used for standardizing Raman instruments and analyzers to give precise spectra in terms of intensity.

Use of the Calibration Tool 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.

2.3 Electrical 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:

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. Keep the door closed when not carrying out service and maintenance work.

⚠ CAUTION

Activities while the Calibration Tool is in operation introduce risk of exposure to measured materials.

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

2.5 Product safety

The Calibration Tool is 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 Rxn analyzers must comply with the applicable safety standards.

2.6 Important safeguards

- Do not use the Calibration Tool for anything other than its intended use.
- Do not open the housing of the Calibration Tool.
- Do not look directly into the laser beam.
- Do not point a laser at a mirrored surface.
- Do not leave attached and unused probes uncapped or unblocked.
- Always use a laser beam block.

2.7 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 Tool (RCT) is used for standardizing Raman instruments and analyzers to give precise spectra in terms of 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 RCT was created specifically for use with Raman instruments and analyzers manufactured by Endress+Hauser.

The RCT contains an intensity reference glass housed in a robust enclosure. The calibration head contains discrete positions for storing, loading, calibration and capturing probe background.

For intensity standardization, a NIST SRM fluorescent glass provides a factory-characterized spectral output. The primary source used in the certification process is a NIST traceable source. A wavelength specific SRM is required for each Raman excitation wavelength.

NOTICE

- ▶ All Endress+Hauser Raman analyzers have built-in wavelength and laser wavelength calibration.

A GRAMS data file (.spc format) characterizing the spectral output of the calibrator is provided on the hard drive of the analyzer or 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 optical components.
- 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 RCT is the ability to capture the probe's background signal. This is used to account for probe to probe background variation when standardizing an instrument.

The standard RCT interfaces with ½ inch probe tips. Please consult the manufacturer for non-standard adapters.

The RCT is moisture sensitive and should be stored in a low humidity environment (see specification section) to avoid degradation of the fluorescent glass. The RCT includes a humidity indicator for a visual reference of its current humidity level.

A software file detailing the spectral characteristics of the device, as well as an electronic certificate, accompanies each unit on a memory stick when new and when recertified.

NOTICE

- ▶ Endress+Hauser recommends recertification after 12 months to ensure that degradation to moisture has not occurred, unless noted differently for a specific application.

CAUTION

- ▶ The RCT should not be installed on a probe unless the probe is at ambient temperature. See *Specifications* for more information. For cryogenic applications this typically takes 30 minutes from the time that the probe has been removed from the cryogenic process. Failure to do so can cause incorrect calibrations and possible damage to the RCT.

CAUTION

- ▶ The probe tip should be cleaned prior to use with the RCT. Failure to do so can cause incorrect calibrations and possible damage to the RCT.

3.1 RCT unit

The front view of the RCT is shown below.

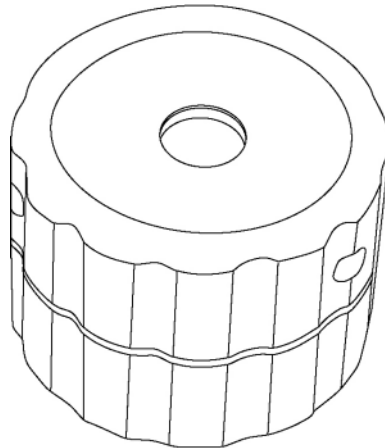


Figure 1. Front view of the RCT unit

The rear view of the RCT contains a window to view the indicating desiccant. If the desiccant is blue, the RCT can be safely used. If the desiccant is tan, then the RCT needs to be sent back to the manufacturer for service and should not be used for calibration purposes.

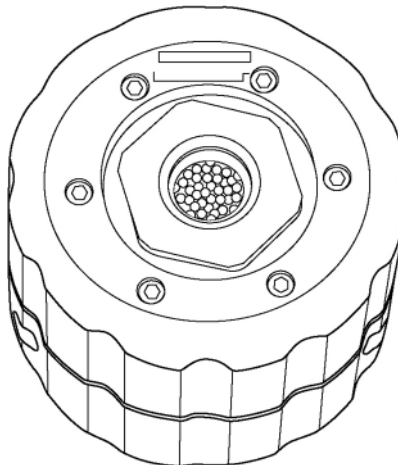


Figure 2. Rear view of the RCT unit

The side view of the RCT, showing the mode selection indicator on the unit.

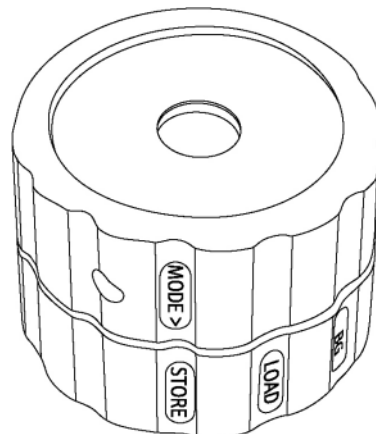


Figure 3. Side view of the RCT unit

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. 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 RCT body.
- In the delivery documents.

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

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

5 Operation

5.1 Mode selection

Mode selection is accomplished by slightly pulling the two parts of the unit apart and rotating the two relative to each other until the mode selection arrow is pointing to the desired mode.

5.1.1 Store

This mode is used for storage as the dust cover will slide into place to protect the unit. The STORE mode moves the dust cover into place. Use this mode to protect the unit from damage when working with probes and/or the calibration accessory.

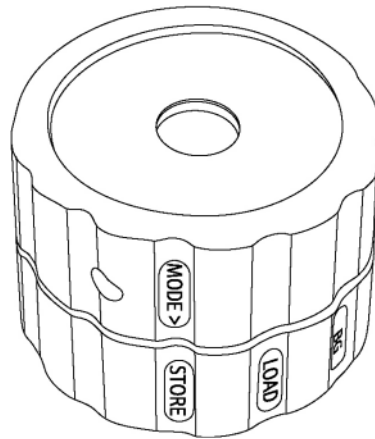


Figure 4. Mode selection: STORE

5.1.2 Load

This mode is used for loading the unit onto a probe. A slight force may be necessary to gently load the unit until it bottoms out on the probe.

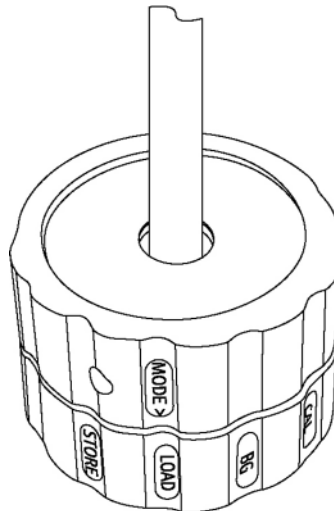


Figure 5. Mode selection: LOAD

NOTICE

- ▶ The RCT should only be installed and removed from a probe in the LOAD position.

5.1.3 BG

The BG mode presents a low Raman signature beam dump for collecting the probe background.

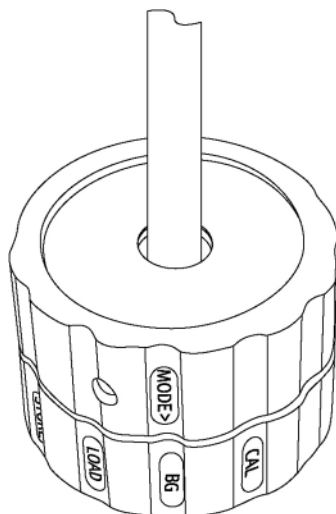


Figure 6. Mode selection: BG

NOTICE

- ▶ The RCT should be properly installed on a probe using the LOAD mode prior to adjusting it to the BG mode.

5.1.4 CAL

The CAL mode is used to present the fluorescent glass to the probe.

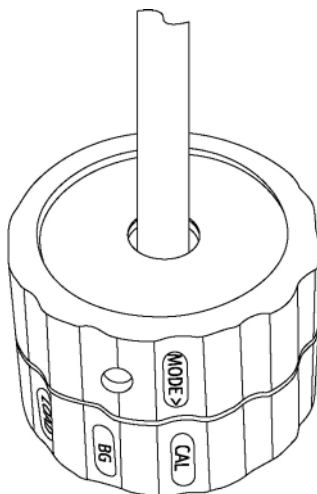


Figure 7. Mode selection: CAL

NOTICE

- ▶ The RCT should be properly installed on a probe using the LOAD mode prior to adjusting it to the CAL mode.

CAUTION

- ▶ The BG and CAL mode apply pressure to the probe to keep it in place and therefore the unit should not be moved in these modes.

5.2 Temperature measurement port

It is necessary to enter the temperature of the device for best results; to aid this measurement a temperature measurement port is part of the unit. A thermometer with the recommended precision and accuracy, compatible with hazardous environments, is provided with the RCT. When inserting the thermometer into the port, thermal grease should be used to ensure a proper thermal interface. Any non-silicone thermal grease is acceptable for use. When complete the thermal grease should be cleaned using cotton swabs or similar.

NOTICE

- ▶ Gloves, such as latex or nitrile, should be worn while working with the thermal paste to avoid contamination.

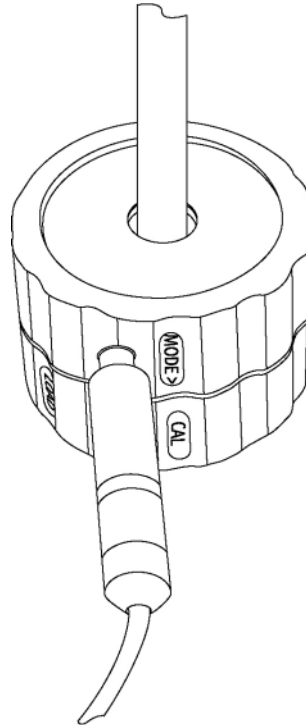


Figure 8. Thermometer inserted into the temperature measurement port

5.3 Humidity indicator

A humidity indicator is incorporated into the RCT to ensure that proper storage conditions are met. The internal desiccant will turn from blue to tan if the internal humidity level exceeds recommended levels. Moisture can degrade the SRM glass over time which can affect the spectral output of the glass. Short intervals of non-condensing, high humidity are acceptable, such as for the duration of a calibration. However, the tool should be kept at a relative humidity level of 10% or less for long-term storage. In the event that the desiccant is tan, the RCT should be returned to the manufacturer for service and should not be used.

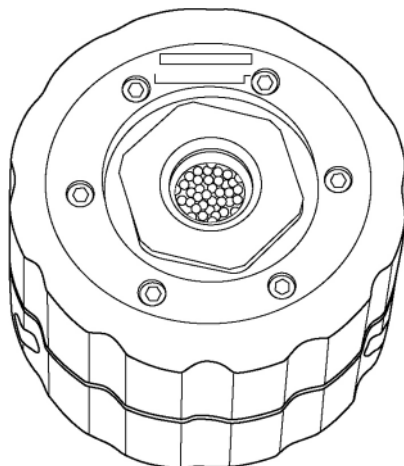


Figure 9. Humidity indicator window

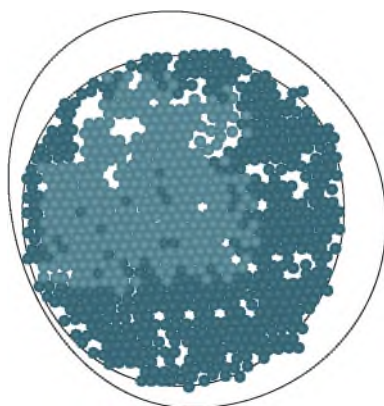


Figure 10. Fully charged desiccant (color BLUE)

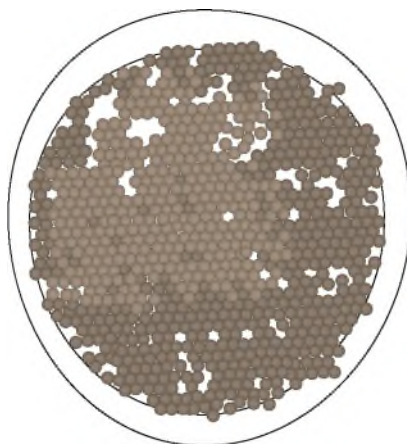


Figure 11. Fully depleted desiccant (Color TAN)

5.4 Desiccant

The RCT ships in a sealed moisture bag with desiccant that is rated for up to 5 years of storage. The RCT should not be removed from this sealed bag until its first use. Once the RCT has been removed from the sealed bag, it should be kept in the sealed storage. An indicating desiccant is provided with the storage case. The desiccant should be removed from its sealed plastic bag upon first use of the RCT. The desiccant will change color from blue to tan once it has been depleted. New desiccant can be purchased as part of the Spare Parts Kit (2017737) for the calibrator. The storage case should always remain closed and sealed except for loading or unloading the RCT. If proper handling and storage are followed, the RCT can remain in the case for up to a year after it has been removed from its sealed bag.

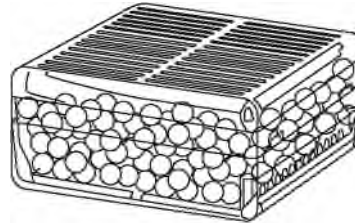


Figure 12. Storage case desiccant

5.5 Moisture protection

The RCT includes a moisture protection bag to be used to protect the unit from direct moisture contact when calibrating in the field. This can be used in both the vertical and horizontal probe positions but is required for the vertical probe position. The bag is first placed onto the probe prior to putting on the RCT. The bag should be positioned such that it fully covers the RCT and then the gland should be hand tightened until it forms a water-tight seal with the probe.

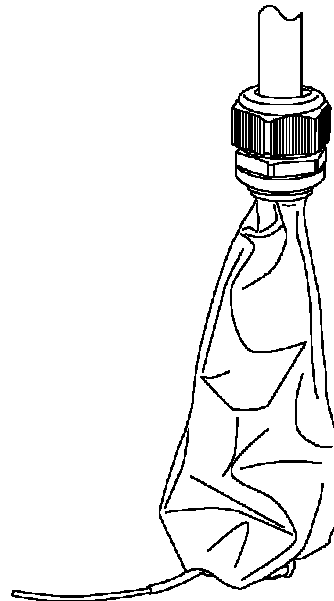


Figure 13. Moisture protect bag in place

6 Maintenance

6.1 Accessory calibration data file

Each RCT is supplied with a source spectral file on a memory stick. The file describes the relative spectral output of the device as measured with the probe tip in contact with the glass. The fluorescent glass inside the unit has a calibration from NIST. However, the sampling characteristics required to match this NIST calibration are atypical to Raman sampling systems. NIST SRM corrections are therefore strongly discouraged when using this device. The source spectral file is a more accurate characterization of the spectral output shape provided by the device when used with an Endress+Hauser system.

It is important to distinguish between calibration of spectral output shape and absolute spectral output level. This device 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 watts/nm (by a factor of λ^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 (cm^{-1}). Therefore, the SSF is in terms of photons per wavenumber.

The software used to operate analyzers is configured to directly read the device’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.

6.2 Raman calibration protocol

The protocol for use of the Raman Calibration Tool 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 (Intensity standard)
3. Probe background standardization (Probe BG)
4. Laser wavelength calibration (Raman shift standard)
5. Calibration qualification (Raman shift standard)
6. Periodic Verification (Verification Service)

A verification standard service is available to perform a periodic calibration verification at the customer’s site. This service uses a secondary calibration device which is used to verify that the spectral calibration of the customer’s RCT is within acceptable tolerance ranges. Please contact your sales or service personnel for additional information.

⚠ CAUTION

- ▶ There is a slight quenching affect for the fluorescent glass and should be allowed to quench for least 2 minutes before you perform the intensity standardization.
- ▶ 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 unit.

7 Technical data

7.1 Specifications

Item	Description
Spectral intensity reference	SRM Fluorescent Glass
Data file spectral range	SRM-532: 534.5 to 694.0 nm
Spectral intensity output repeatability (at time of certification)	< ± 2 %
Total long term spectral uncertainty (at any wavelength)	SRM-532: $\pm 2.85\%$
NIST traceable primary standard uncertainty	Available upon request
Power source	N/A
Power consumption	None
Unit dimensions (Diameter x Height)	83 x 66 mm (3.25 x 2.6 inches)
Unit mass (weight)	0.54 kg (1.2 lb.)
CE certified	N/A
IP Rating	IP10
Operating Conditions	-20 °C to 50 °C, < 95% Humidity Non-Condensing
Recommended Storage Conditions	15 °C to 25 °C < 10% Humidity Non-Condensing
Absolute Maximum Storage Conditions	-20 °C to 60 °C < 10% Humidity Non-Condensing

Table 4. Specifications

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