



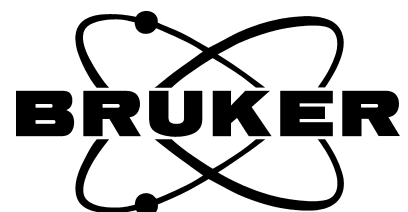
OPUS

Spectroscopy Software

Version 6

User Manual

OVP Raman



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This manual is the original documentation for the OPUS spectroscopic software.

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1 Introduction

This manual describes OVP (OPUS Validation Program) for Raman spectrometers. OVP Raman is an integral part of the OPUS spectroscopic software and is used to test the Bruker line of Raman spectrometers and Raman modules according to the instrument specifications. For further general information on OVP refer to the OPUS Reference Manual.

1.1 PQ Test Protocol

The Performance Qualification (PQ) test protocol is a short-term test which evaluates the spectroscopic performance and proper function of the Raman spectrometer. To ensure that the instrument is working properly the PQ test protocol is intended to be run on a day-by-day basis.

The following single tests can be run during PQ:

- Spectral characteristics test
- Wavelength accuracy test
- Resolution test
- Laser power test
- Response intensity test
- Signal-to-noise test

1.2 OQ Test Protocol

The Operational Qualification (OQ) test protocol is a test which checks the instrument performance and compares this performance to the instrument specifications. In general, the OQ test protocol is performed during final testing before an instrument is delivered to the customer. The OQ test should be performed after each major repair, exchange of optical components which may influence the instrument performance, and on a regular yearly basis after maintenance has taken place.

The following single tests can be run during OQ:

- Spectral characteristics test
- Cut off test
- Signal-to-noise test
- Wavelength accuracy test
- Resolution test
- Noise test

2 OVP Test Setup

To be able to perform the different OVP for Raman tests a set of reference standards is required. The following table shows test and standards which are used for the different tests. Make sure that the required standards are available.

Instrument Type	Tests	Standard	Source
RFS 100 FRS 106 RAM II RamSys	Laser Wavenumber Calibration Spectral Characteristics Test Cut off Test	Spectralon	White light source
RFS 100 FRS 106 RAM II	Wavelength Accuracy Test Resolution Test	Naphthalene sample	Lase
RamSys	Wavelength Accuracy Test Resolution Test	Polystyrene sample	Laser
RFS 100 FRS 106 RAM II RamSys	Signal to Noise Test Laser Power Test Response Intensity Test	Polystyrene sample	Laser
RFS 100 FRS 106 RAM II RamSys	Noise Test	No sample	No source

Before you can use OVP for Raman you have to set up the OVP test channels and OVP tests. Select the *OVP Setup* command from the *Validation* menu to open the following dialog:

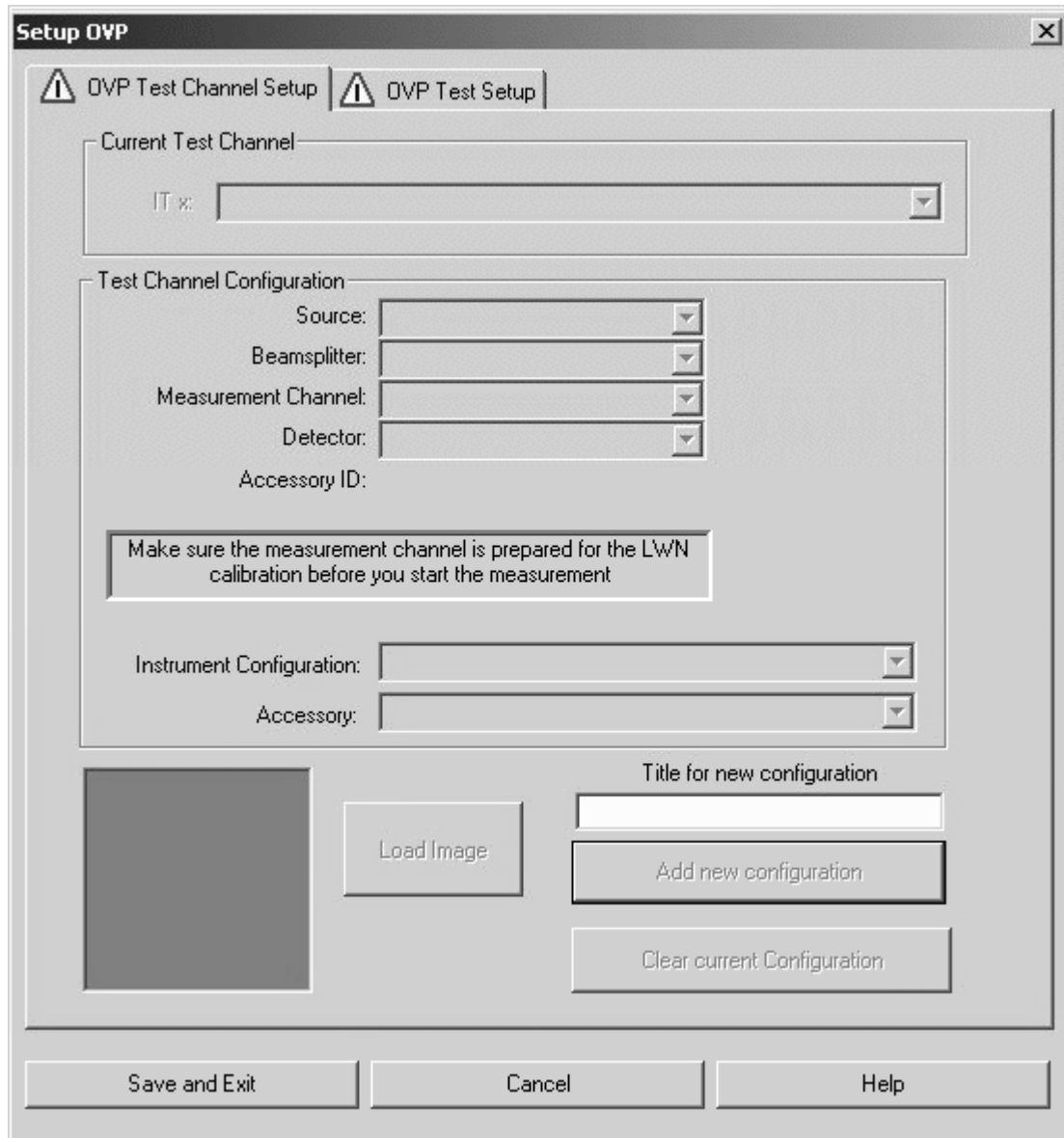


Figure 1: Setup OVP

Enter a title for the current configuration into the *Title for new configuration* entry field, to specify the current setup by a descriptive text.

Click on the *Add new configuration* button. Now, all the other selection fields will no longer be hidden and can be filled in.

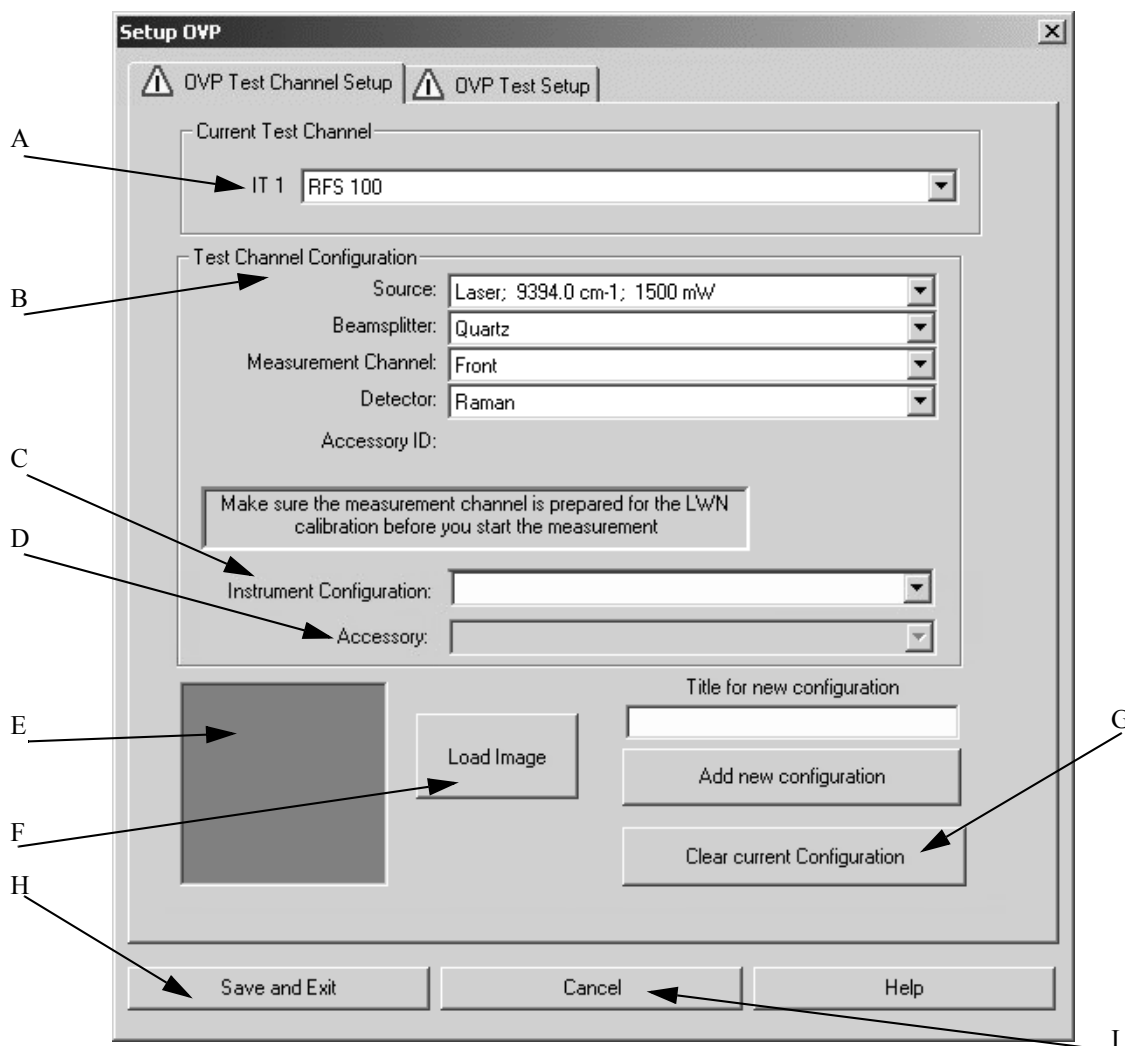



Figure 2: Setup OVP for Raman - Activated drop-down lists

- A) The drop-down list shows the test channel used by OVP.
- B) For each spectrometer test channel you have to assign a measurement configuration to be used during actual validation measurement. This is a valid combination of source, beam splitter, measurement channel and detector. As soon as you have selected all components the appropriate instrument configuration (C) and the bitmap (E) specified for the measurement channel will automatically be displayed.
- C) Select the Raman configuration from the *Instrument Configuration* drop-down list.
- D) At present, Raman accessories cannot be selected from the drop-down list.
- E) Depending on the measurement channel selected a configuration-specific bitmap will be displayed.
- F) Use the *Load Image* button to load further instrument configurations available in OPUS. The bitmaps are stored in the *OPUS\VALIDATION\Bitmaps* directory. Select the *Thumbnail* option from the pop-up menu when clicking on the  button to be able to see at once which bitmap belongs to which file name (see also OPUS Reference Manual, chapter 13). You can also make your own photo with a size of 90 x 90 and store it into the bitmap directory using the *ITnn.bmp* file name (*nn* is a consecutive number).
- G) To delete the current configuration click on this button. Once deleted the settings cannot be reloaded. If you have accidentally deleted a channel, you have to repeat the complete setup again. OVP configurations are stored in the *OPUS\VALIDATION\Database* directory.

- H) To save the configuration click on the *Save and Exit* button.
- I) Click on this button to interrupt the setup without saving.

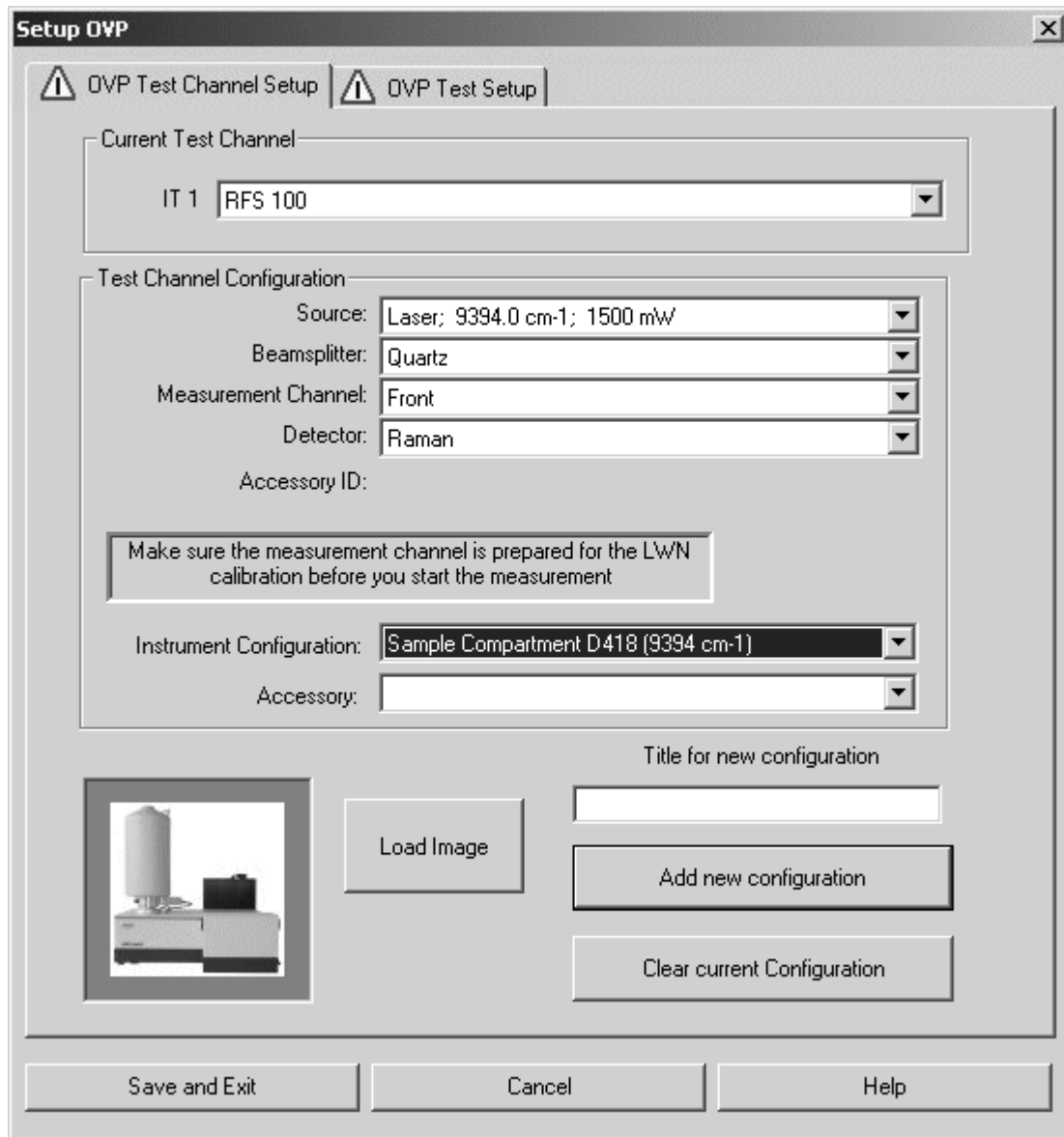


Figure 3: Setup OVP for Raman - Bitmap of instrument type

If you click on the *OVP Test Setup* tab, the following dialog opens:

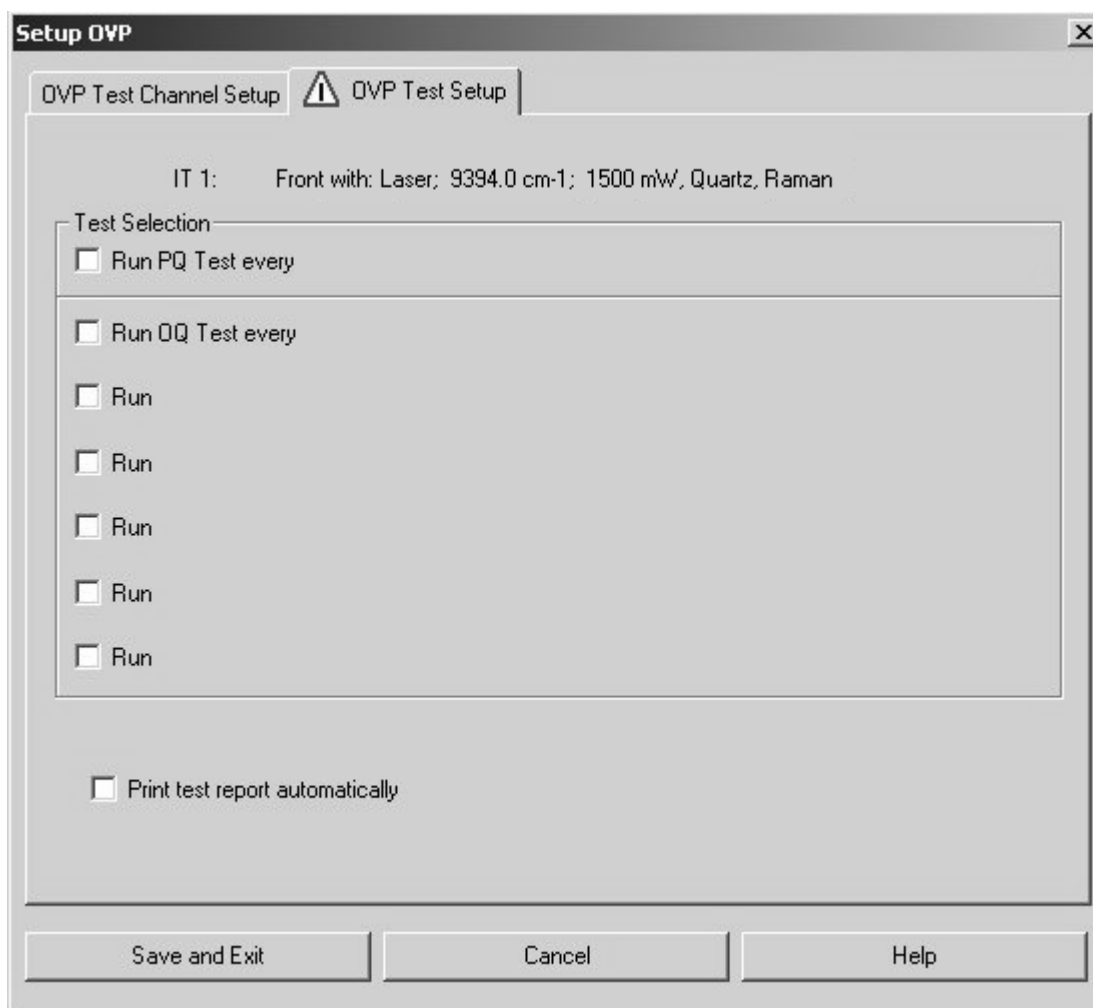


Figure 4: OVP Test Setup tab for Raman

The line on the very top shows the test channel selected on the *OVP Test Channel Setup* tab.

You have two basic categories for testing:

- PQ test
- OQ test

Define a specific test interval for both test categories to determine when the tests have to be performed.

Note: It is recommend running *PQ* tests as a short-term test on a daily basis (24 hours). *OQ* tests should normally run after installation, maintenance or major repair, e.g. every 6 or 12 months.

2.1 PQ Tests

Activate the *Run* check box to have displayed the following line:

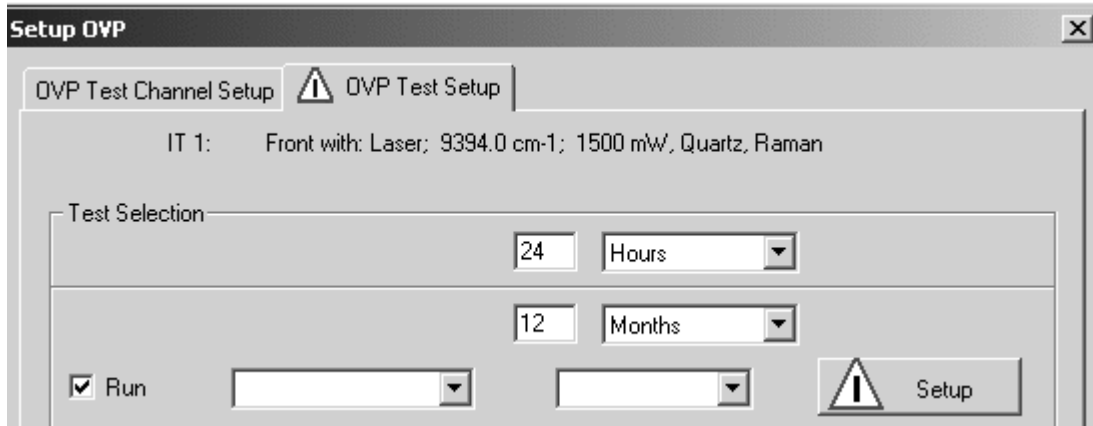
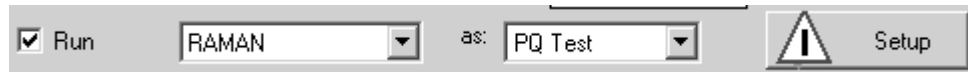


Figure 5: Configuring Raman test

Select *RAMAN* from the first drop-down list and the PQ test category from the second. Type the interval period into the entry field and select the units from the drop-down list. Available units are hours, days, or months.



Click on the *Setup* button to open the following dialog:

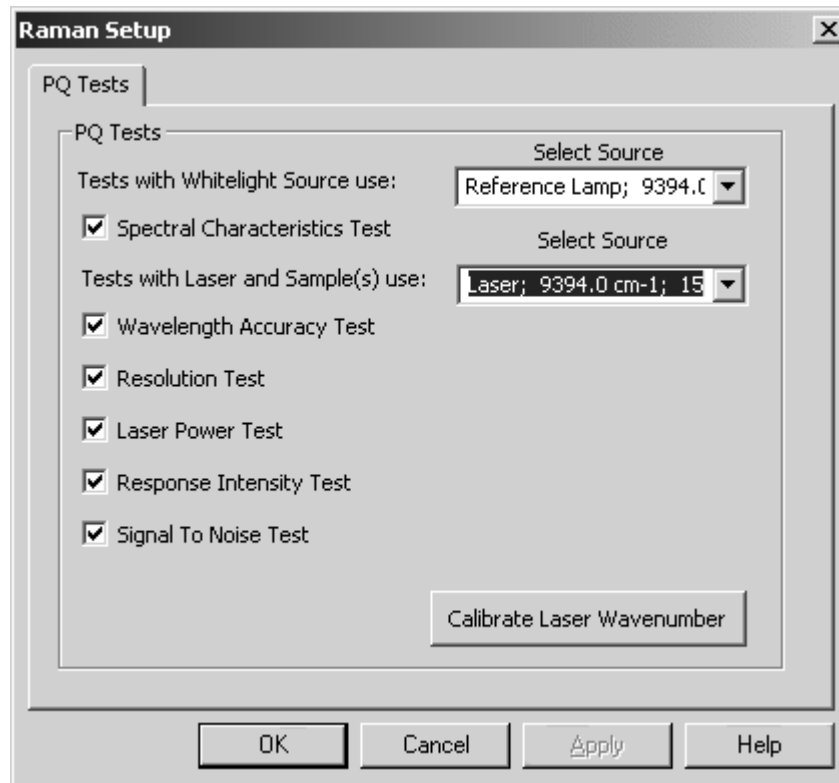


Figure 6: PQ test setup for Raman

All tests available for a particular type of spectrometer have already been selected. In exceptional cases (not recommended) you can deactivate certain tests by deleting the check mark.

Select the respective source from the *Select Source* drop-down list (see also the table at the beginning of chapter 2).

Tests which are not available for the given spectrometer type cannot be selected.

If you click on the *OK* button, the current setup status is indicated by a green check mark on the *Setup* button.

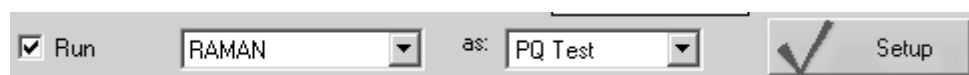


Figure 7: PQ test status for Raman

2.1.1 Calibrating Laser Wavenumber

To measure the laser wavenumber for the currently selected IT channel click on the *Calibrate Laser Wavenumber* button. In OPUS it is possible to store and use individual laser wavenumber values for each test channel defined. For further details on the laser wavenumber see the OPUS Reference Manual.

The calibration is no OQ/PQ test and is used to check and set the laser wavenumber. To perform the calibration use the white light source.

Test description:

- 1) Before starting the laser wavenumber calibration you have to place the spectralon reflector onto the measuring position.
- 2) Click on the *Calibrate Laser Wavenumber* button (figure 6). Note that this button is accessible in both OQ and PQ setup dialogs.
- 3) Based on the water vapor absorption band at 7306.74cm^{-1} the HeNe laser wavenumber is determined and the result displayed.
- 4) If the laser wavenumber is within the specified limit, you can exit the dialog. If it is beyond the specification, set the laser wavenumber again by clicking on the *OK* button.

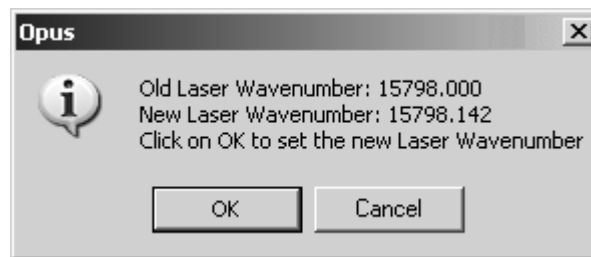


Figure 8: OVP for Raman - Result dialog of laser wavenumber calibration

The single PQ tests which can be run when working with a Raman instrument are described in the following.

2.1.2 Spectral Characteristics Test

This test characterizes the overall spectral response of the Raman instrument. The test is performed by using a white light source. Before starting the spectral characteristic test you have to place the spectralon reflector onto the measuring position.

Note: Make sure that the light emitted from the white light source is correctly reflected into the direction of the collecting lens by the spectralon reflector.

Test description

- 1) A single-channel spectrum is measured by using the parameters from the database.
- 2) Define the maximum spectral value at maximum and left/right edge. The determination of intensities on the left and right edge refers to a range of 20cm^{-1} around the specified frequency. The values will be evaluated according to the following equation:

$$\text{Value}_{l/r} = \text{Intensity}_{l/r} / \text{Maximum}_{l/r} \text{ (left or right edge)}$$
- 3) The test has passed if the spectral characteristic values, which have been evaluated, exceed instrument-specific values.

2.1.3 Wavelength Accuracy Test

This test is used to check the wavelength accuracy of the Raman instrument. The test is performed by using a naphthalene or polystyrene test sample in case of RamSys. Before starting the spectral resolution test you have to place the naphthalene (polystyrene) test sample onto the measuring position.

In case of naphthalene deposit the brass disc with the positioned glass window towards the collecting lens assembly. In case of polystyrene place a flat mirror (use the one provided by the accessory kit) behind the polystyrene sample. Select the *Check Signal* tab from the *Measurement* dialog in the *OPUS Measure* menu to maximize the signal.

Test description

- 1) A Raman spectrum is measured by using the parameters from the database.
- 2) The following bands are determined for evaluation:
 - $513.8\text{cm}^{-1} \pm 1.5\text{cm}^{-1}$ (naphthalene)
 - $1382.2\text{cm}^{-1} \pm 1.5\text{cm}^{-1}$ (naphthalene)
 - $3056.4\text{cm}^{-1} \pm 1.5\text{cm}^{-1}$ (naphthalene)
 - $1001.4\text{cm}^{-1} \pm 0.54\text{cm}^{-1}$ (polystyrene)
- 3) The test has passed if the deviations are less than the maximum deviation specified for each band.

2.1.4 Resolution Test

The spectral resolution test is performed to check the achievable spectral line width of a Raman spectrum. The test is performed by using a naphthalene test sample (polystyrene for RamSys) which has already been placed onto the sample position of the previous wavelength accuracy test. If available, the spectrum from the previous measurement is used.

Test description

- 1) Either the spectrum from the wavelength accuracy test is used or a measurement is performed by using the parameters from the database.
- 2) The half-width of the Raman band at 513.8cm^{-1} (naphthalene) or 1001.4cm^{-1} (polystyrene) is obtained from the band table (parameter width).
- 3) The test has passed if the half-width is less than the resolution specified.

2.1.5 Laser Power Test

This test is used to check the proper operation mode of the Raman laser. The test is performed by using a polystyrene test sample. Before starting the laser power test you have to place the polystyrene test sample onto the measuring position. The sample has to be backed by the flat mirror provided by the Raman accessory kit.

Test description

- 1) Two measurements are performed by using the parameters from the database. The laser power value (low) of the first measurement is obtained from the measurement parameters. The laser power value (high) of the second measurement corresponds to 80% of the maximum laser power.
- 2) The peak band position at 1001.4cm^{-1} for both spectra is determined by using the *Peak Pick* command from the *Evaluate* menu.
- 3) The band positions of both measurements must not deviate from each other by more than the value specified.

2.1.6 Response Intensity Test

This test is used to check the photometric accuracy of subsequent measurements. The test is performed by using a polystyrene test sample. Before starting the response intensity test you have to place the polystyrene test sample onto the measuring position.

Test description

- 1) Five subsequent measurements are performed by using the parameters from the database.
- 2) The peak maximum is determined between 1000cm^{-1} and 1002cm^{-1} . The average intensity is determined for all spectra, and the maximum deviation from the average of the values is calculated.
- 3) The maximum deviation should not exceed the maximum percentage value specified.

2.1.7 Signal-To-Noise Test

The signal-to-noise ratio is determined to check the sensitivity of the instrument. The test is performed by using a polystyrene test sample. Before starting the signal-to-noise test you have to place the polystyrene test sample onto the measuring position.

Additionally, place a flat mirror (use the one provided by the accessory kit) behind the polystyrene sample. Select the *Check Signal* tab from the *Measurement* command in the *Measure* menu to maximize the signal.

Test description

- 1) One measurement is performed by using the parameters from the database.
- 2) The maximum peak level is determined between 1000cm^{-1} and 1002cm^{-1} and used as reference value for the signal.
- 3) The noise is determined between 2000cm^{-1} to 2100cm^{-1} by using the *Signal-to-Noise Ratio* command with peak-to-peak mode.
- 4) The signal-to-noise value is the ratio of the signal from 2) and the noise from 3).
- 5) The test has passed if the signal-to-noise is greater than the instrument-specific value.

2.2 OQ Tests

The OQ test protocol is a test which checks the instrument performance and compares it to the instrument specifications. The OQ test protocol is generally performed during final testing before an instrument is delivered to the customer. The OQ test should be performed after each major repair, exchange of optical components which may influence the instrument performance, and on a regular yearly basis, e.g. after maintenance.

If you activate the *Run* check box on the *OVP Test Setup* tab, the following line will be displayed:

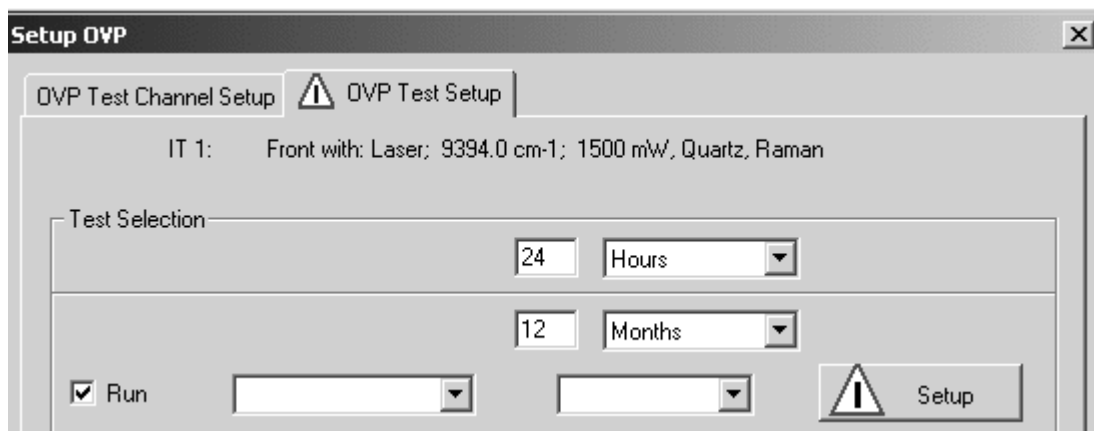
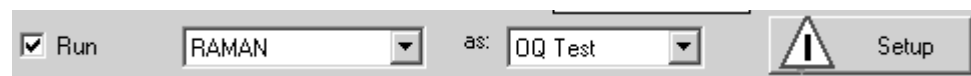


Figure 9: Configuring Raman test

Select *RAMAN* from the first drop-down list and the PQ test category from the second. Type the interval period into the entry field and select the units from the drop-down list. Available units are hours, days, or months.



Click on the *Setup* button to open the following dialog:

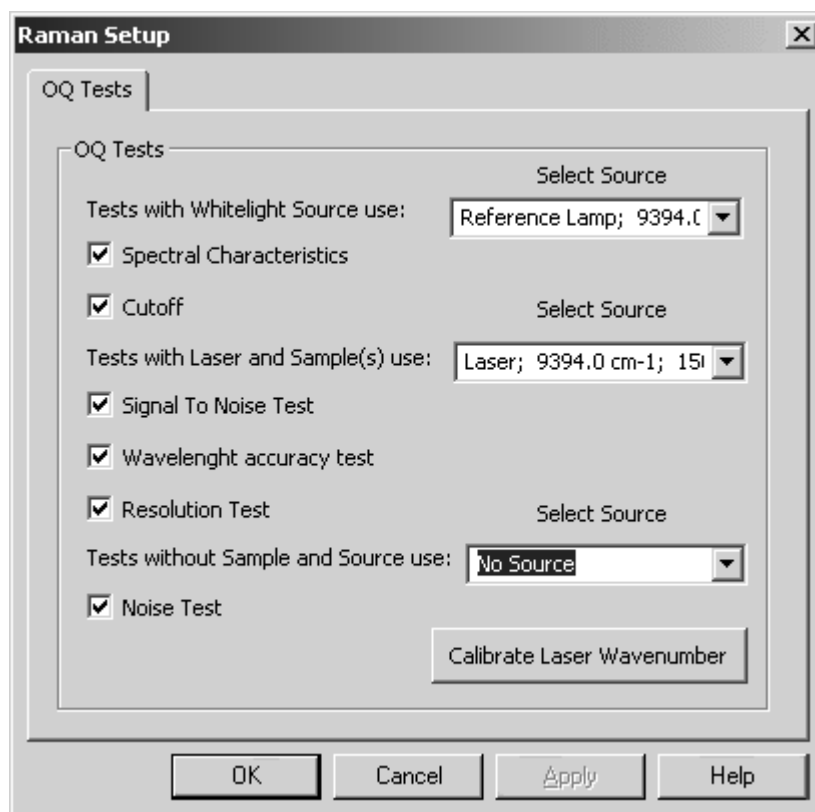


Figure 10: OQ test setup for Raman

Tests which are not available for the selected instrument type are disabled, all the other tests are activated. If you do not want to perform a specific test, deactivate the respective check box. Select the respective source from the *Select Source* drop-down list (see also the table at the beginning of chapter 2).

As soon as you have set up all tests click on the *OK* button. The current setup status is indicated by a green check mark on the *Setup* button of the *OVP Test Setup* tab.

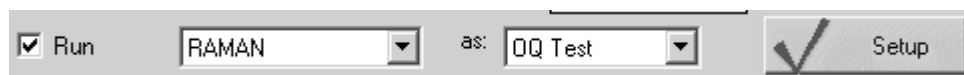


Figure 11: OQ test status for Raman

The single OQ tests which can be run when working with a Raman instrument are described in the following.

2.2.8 Spectral Characteristics Test

This test characterizes the overall spectral response of the Raman instrument. The test is performed by using the white light source. Before starting the spectral characteristic test you have to place the spectralon reflector into the measuring position.

Note: Make sure that the light emitted from the white light source is correctly reflected into the direction of the collecting lens by the spectralon reflector.

Test description

- 1) A single-channel spectrum is measured according to the parameters in the database.
- 2) Define the maximum spectral value at maximum and left/right edge. The determination of intensities on the left and right edge refers to a range of 20cm^{-1} around the specified frequency. The values will be evaluated according to the following equation:
$$\text{Value}_{l/r} = \text{Intensity}_{l/r} / \text{Maximum} \text{ (}_{l/r}\text{: left or right edge)}$$
- 3) The test has passed if the spectral characteristic values, which have been evaluated, exceed instrument-specific values.

2.2.9 Cutoff Test

The test determines the low detector frequency limit. If available, the spectrum from the spectral characteristics test is used.

Test description

- 1) To calculate the low frequency limit the maximum signal (noise) is determined between instrument-specific frequency limits.
- 2) The cutoff frequency in the spectrum is the position where the intensity is 1.5 times greater than the value of the noise level determined in 1).
- 3) The test has passed if the cutoff frequency is above an instrument-specific value.

2.2.10 Signal-To-Noise Test

The signal-to-noise ratio is determined to check the sensitivity of the instrument. The test is performed by using a polystyrene test sample. Before starting the signal-to-noise test you have to place the polystyrene test sample onto the measuring position.

Additionally, place a flat mirror (use the one provided by the accessory kit) behind the polystyrene sample. Select the *Check Signal* tab from the *Measurement* command in the *Measure* menu to maximize the signal.

Test description

- 1) Ten measurements are performed by using the parameters from the database.
- 2) The maximum peak level between 1000cm^{-1} and 1002cm^{-1} is determined and used as reference value for the signal.
- 3) The noise between 2000cm^{-1} to 2100cm^{-1} is determined by means of the *Signal-to-Noise Ratio* command with peak-to-peak mode.
- 4) The signal-to-noise value is the ratio of the signal from 2) and the noise from 3).
- 5) The test has passed if the average of the signal to noise values of all 10 measurements is greater than the instrument-specific value.

2.2.11 Wavelength Accuracy Test

This test is used to check the wavelength accuracy of the Raman instrument. The test is performed by using a naphthalene or polystyrene test sample in case of RamSys. Before starting the spectral resolution test you have to place the naphthalene (polystyrene) test sample onto the measuring position.

In case of naphthalene deposit the brass disc with the positioned glass window towards the collecting lens assembly. In case of polystyrene place a flat mirror (use the one provided by the accessory kit) behind the polystyrene sample. Select the *Check Signal* tab from the *Measurement* dialog in the *OPUS Measure* menu to maximize the signal.

Test description

- 1) A Raman spectrum is measured by using the parameters from the database.
- 2) The following bands are used for evaluation:
 - $513.8\text{cm}^{-1} \pm 1.5\text{cm}^{-1}$ (naphthalene)
 - $1382.2\text{cm}^{-1} \pm 1.5\text{cm}^{-1}$ (naphthalene)
 - $3056.4\text{cm}^{-1} \pm 1.5\text{cm}^{-1}$ (naphthalene)

 - $1001.4\text{cm}^{-1} \pm 0.54\text{cm}^{-1}$ (polystyrene)
- 3) The test has passed if the deviations are less than the maximum deviation specified for each band.

2.2.12 Resolution Test

The spectral resolution test is performed to check the achievable spectral line width of a Raman spectrum.

Note: The spectral resolution test is specified for routine analysis. The spectral resolution depends on the type of system and can be between 2cm^{-1} and 8cm^{-1} . If required, a special test for a spectral resolution of 1cm^{-1} for special instruments is also available on request. This special test will be run apart from OVP by Bruker Service.

The test is performed by using a naphthalene or polystyrene test sample which has already been placed onto the sample position of the previous wavelength accuracy test. If available, the spectrum from the previous measurement is used.

Test description:

- 1) Either the spectrum from the wavelength accuracy test is used or a measurement is performed by using the parameters from the database.
- 2) The half-width of the Raman band at 513.8cm^{-1} (naphthalene) or 1001.4cm^{-1} (polystyrene) is obtained from the peak table (parameter width).
- 3) The test has passed if the half-width is less than the resolution specified.

2.2.13 Noise Test


The noise test checks whether the noise level of the Raman detector is at an appropriate value and whether the noise spectrum is free from artifacts caused by external light emission.

The test is performed without using any sample, and with the source being switched off.

Test description

- 1) One measurement is performed by using the parameters from the database.
- 2) Determine the peak-to-peak noise between 0cm^{-1} and 3500cm^{-1} .
- 3) The test has passed if the noise does not exceed a maximum limit defined.

3 Run OVP Tests

There are two possibilities to run OVP tests. Either by selecting the Run *OVP Tests* command in the *Validation* menu or by clicking on the status light () and using the *Instrument Status* dialog. For details on the letter case refer to the OPUS Reference Manual, chapter 13.

To start running the tests click on the *Run OVP Tests* command in the *Validation* menu. The following dialog opens:

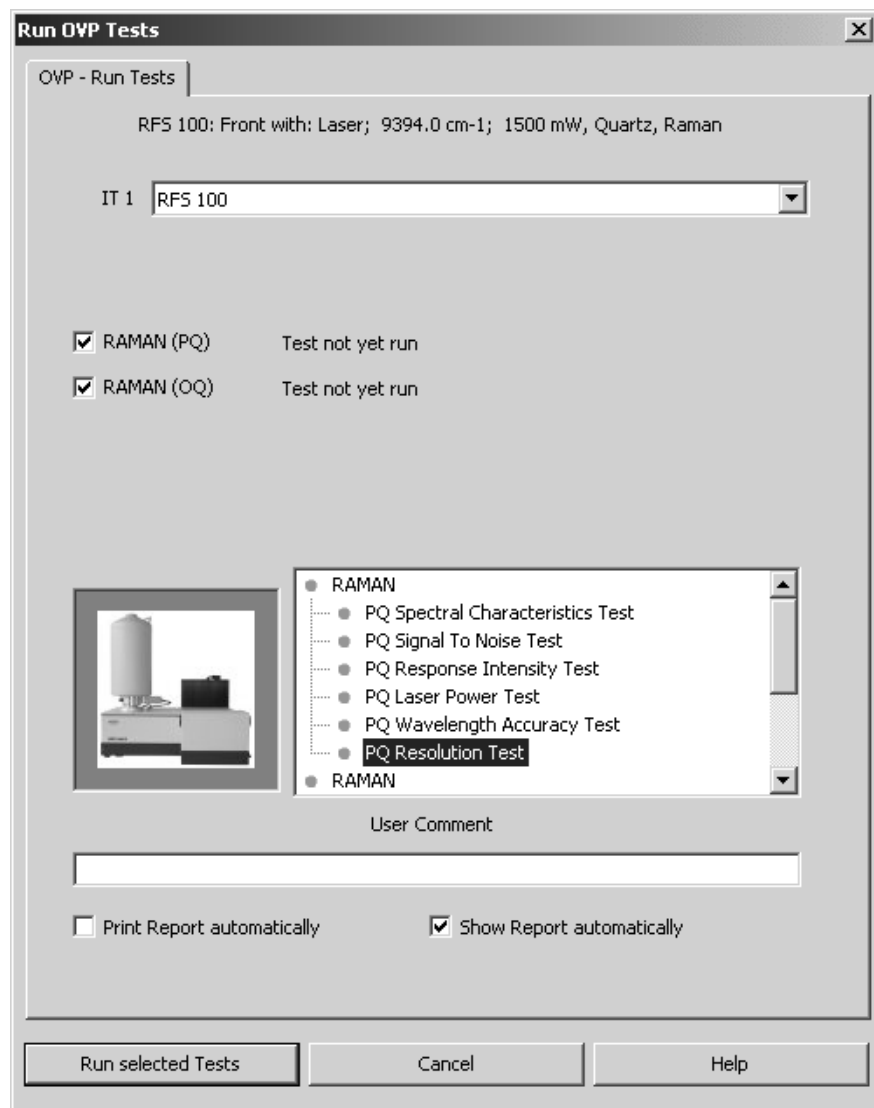


Figure 12: Run OVP Tests

The *ITn* drop-down list allows to select the appropriate IT test channel. The lines below show the tests set up for the IT test channel selected, as well as the test status. Tests which interval has expired, or which have not yet been performed are automatically activated for measurement. Tests which interval has not yet expired are not activated, and their remaining time is indicated as a reminder.

If a test has not expired, but the interval has only e.g. 2 hours left you can activate the check box for that test and run the test earlier.

If a test has expired, but you are not prepared to perform that test (external standards required), you can deactivate the check box for the corresponding test.

Note: If you decide not to perform a test which has already been expired, you will not be able to use this channel in routine environment.

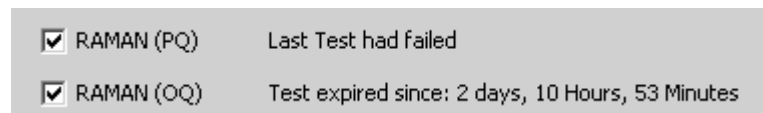


Figure 13: Different types of OVP test status

As soon as you have activated the check box for the respective test protocol you can see which test(s) will be performed by the browser-like display next to the bitmap.

All measurement results are stored in a report (PDF file) and recorded in the *UserData* database. To append a comment to the current report write a comment into the *User Comment* entry field.

Click on the *Run selected Tests* button to start the respective tests. If you do not want to perform any test, click on the *Cancel* button.

3.1 Printing Reports

The results of test measurements are stored in a report (PDF format). The test report contains a summarized test result page and subsequent pages for each test with all the test spectra displayed. Reports are stored in the separate *VALIDATION\REPORTS* directory and can automatically be displayed and/or printed. To have the report displayed and printed you require the Adobe Acrobat Reader which is available on the OPUS installation CD.

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