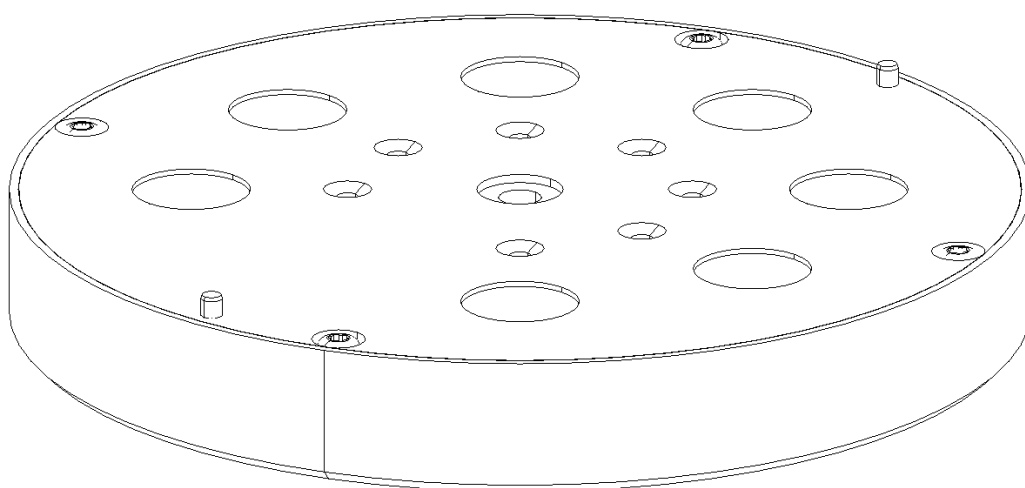




USP Performance Verification Kit

Operation Manual



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BUCHI reserves the right to make changes to the manual as deemed necessary in the light of experience; especially in respect to structure, illustrations and technical detail.

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Table of contents

1	About this manual	5
2	Safety.	6
2.1	Warning notices used in this manual	6
2.2	General safety rules	6
3	Technical data	7
3.1	Scope of delivery for reflection measurement	7
3.1.1	USP Standards	7
3.1.2	CD containing Standard Certificate file	8
3.1.3	Hardcopy of the Standard Certificate	8
3.1.4	Add-on for measuring cells for reflection measurement	8
3.2	Scope of delivery for solid transmission measurement.	9
3.2.1	USP Standards	10
3.2.2	CD containing Standard Certificate file	10
3.2.3	Hardcopy of the Standard Certificate	10
3.4	Activating the USP test kit license	11
4	Description of the standards and the test.	13
4.1	Introduction	13
4.2	Photometric linearity test	13
4.3	Noise test	13
4.4	Wavenumber accuracy	14
4.5	Wavenumber repeatability	14
5	Overview over the steps to be carried out.	15
6	SOP for performing the USP regular test	16
6.1	Preparation	16
6.2	Working with several USP kits in one database	17
6.3	Description of the main window in the NIRWare Operator	18
6.3.1	Menu structure service menu	18
6.3.2	Toolbar	18
6.3.3	Info area.	18
6.3.4	Iconbar for graphic and report.	18
6.3.5	Graphic and report area.	19
6.3.6	Test value area - Overview.	19
6.3.7	Iconbar for test values and spectra	19
6.3.8	Iconbar for graphics	19

Read this manual carefully before installing and running the USP test and note the safety precautions in chapter 2 in particular. Store the manual in the immediate vicinity of the instrument, so that it can be consulted at any time.

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6.4	Positioning the standards	20
6.4.3	NIRFlex Solids Transmittance	21
6.4.4	NIR Checkmaster	21
6.5	Starting the test	22
6.5.1	Reflection measurement.	22
6.5.2	Solid Transmission measurement	23
6.6	Evaluating the tests	27
6.6.1	Linearity test.	27
6.6.2	Noise test	33
6.6.3	Wavelength test	37
6.7	Printing and signing the test data	39
6.7.1	Printing spectra	39
6.7.2	Printing the USP Protocol	39
7	Maintenance.	41
7.1	Cleaning.	41
7.2	Recertification of the USP test kit	41

1 About this manual

This manual describes the Performance Verification Test Kit for the NIRFlex N-500 in the pharmaceutical industrial environment and provides all information required for its safe execution and to maintain the test kit in good working order.

In particular laboratory personnel and operators are addressed.

The Performance Verification Test Kit operation manual Version A is created according to the latest version of NIRWare.

NOTE:

The symbols pertaining to safety (WARNINGS and ATTENTIONS) are explained in chapter 2.

Abbreviations

BREOS: Buchi Rare Earth Oxides Standard

GS: Gray standard

RMS: Route Mean Squares

USP: United States Pharmacopeia

Ph.EUR: European Pharmacopeia

2 Safety

This chapter points out the safety concept of the USP Performance Verification Kit and contains general rules of behavior and warnings from hazards pertaining to the use of the kit. The safety of users and personnel can only be ensured if these safety instructions and the safety-related warnings in the individual chapters are strictly observed and followed. Therefore, the manual must always be available to all persons performing the tasks described herein.

2.1 Warning notices used in this manual



WARNING

Generally, the triangular warning symbol indicates the possibility of personal injury or even loss of life if the instructions are not followed.



ATTENTION

With the general “Read this” symbol, ATTENTIONs indicate the possibility of equipment damage, malfunctions or incorrect process results, if instructions are not followed.

NOTE:

Useful tips for an easy operation of the instrument.

2.2 General safety rules

Duty of maintenance and care

The user is responsible for ensuring that the instrument is operated in proper condition only, and that maintenance, service, and repair jobs are performed with care and on schedule, and by authorized personnel only.

Modifications

Modifications to the USP Performance Verification Kit are only permitted after prior consultation with and with the written approval of the manufacturer. Modifications and upgrades shall only be carried out by an authorized Buchi technical engineer. The manufacturer will decline any claim resulting from unauthorized modifications.

3 Technical data

This chapter contains technical data, requirements and performance data of the USP Performance Verification Kit.

The USP test kit for reflection measurement is produced at the Buchi production site and tested with the master NIRFlex N-500 and the measuring cell NIRFlex Solids. The master system is validated using the standard reference material® (SRM NIST 1920a).

The USP test kit for solid transmission measurement is produced at the Buchi production site and tested with the master NIRFlex N-500 and the measuring cell NIRFlex Solids Transmittance. The master system is validated using the standard reference material® (SRM NIST 2065).

3.1 Scope of delivery for reflection measurement

All items belonging to the USP Performance Verification Kit are delivered in a special box.

For reflection measurement (NIRFlex Solids and NIRFlex Fiber Optics Solids): N512-002

3.1.1 USP Standards

The Buchi USP Standards are integrated in a disk. This disk contains one white standard, 5 gray standards and 1 standard (Buchi Rare Earth Oxide Standard) for checking the wavenumber accuracy and repeatability. All standards are protected with a window of special glass.

Table 2-1: Technical data of reference material for reflection measurement

Standard ID	Standard description	Standard composition
White	White standard	Spectralon®
GS 80 %	Gray standard with nominal 80 % reflectivity	Spectralon® with carbon black
GS 40 %	Gray standard with nominal 40 % reflectivity	Spectralon® with carbon black
GS 20 %	Gray standard with nominal 20 % reflectivity	Spectralon® with carbon black
GS 10 %	Gray standard with nominal 10 % reflectivity	Spectralon® with carbon black
GS 5 %	Gray standard with nominal 5 % reflectivity	Spectralon® with carbon black
BREOS	Buchi Rare Earth Oxide Standard	Mixture of rare earth oxides

The General Chapter <1119> of the US Pharmacopeia and Chapter 2.2.40 of the European Pharmacopoeia request the verification of the wavelength uncertainty or scale.

US Pharmacopeia: The “**USP Near-IR System Suitability Reference Standard**”¹ or the “National Institute of Standards and Technology (NIST) **Standard Reference Material (SRM) 2036**”² for reflectance measurements...can be used. Suitable materials for demonstrating wavelength dispersion performance include polystyrene, trichlorobenzene, mixtures of rare earth oxides, and absorption by water vapor for instruments that use an interferometer for wavelength dispersion. With appropriate justification, alternative standards may be used.

1 G.E. Ritchie et al., J. Near Infrared Spectrosc. 16, 205-210 (2008)

2 SRM 2036 has several advantages over NIST 1920a, namely, reduced dependence of band positions on system resolution and well defined temperature coefficients. It uses the same material as NIST's transmission standard SRM 2035. However, most of the absorption bands of SRM 2036 and SRM 2035 are very broad, which makes the determination of the correct band position less reliable. Therefore Buchi does not use these standards.

European Pharmacopoeia: A *mixture of dysprosium, holmium and erbium oxides* (1+1+1 by mass) or other certified material may be used.

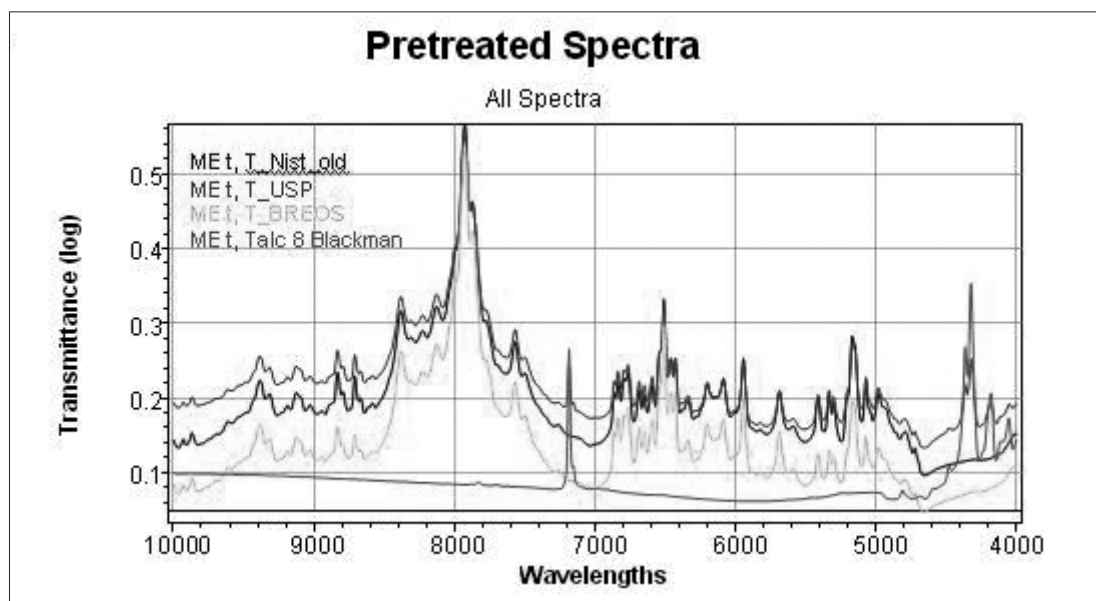


Fig 3.1: Spectra comparison

The Buchi Performance Verification Kit for the NIRFlex N-500 (N512-002) includes the BREOS wavelength standard, which is traceable to NIST SRM 1920a. BREOS is a mixture of dysprosium, holmium and erbium oxides. It also includes a USP Near-IR System Suitability Reference Standard.

The figure above shows the spectra of the NIST SRM 1920a (T_Nist_old), the USP Near-IR System Suitability Reference Standard (T_USP), the BREOS (T_BREOS) and talc (Talc_8_Blackman). All spectra were recorded using a NIRFlex N-500.

The figure clearly demonstrates that all rare earth oxide spectra coincide very well. The USP Near-IR System Suitability Reference Standard contains talc in addition to the rare earth oxides, which gives rise to two additional absorption bands at approximately 7250 cm⁻¹ and 4250 cm⁻¹.

The excellent match of the band positions of the rare earth oxides of the different standards clearly demonstrates the justification for the use of the BREOS wavelength standard for the Performance Verification Kit and its traceability to the NIST 1920a.

3.1.2 CD containing Standard Certificate file

The Standard Certificate file is delivered in xml-format and contains the expected values (% reflectance and wavenumbers for reflection measurement) of the standards in relation to the master used for measurement at the Buchi production site before shipment.

3.1.3 Hardcopy of the Standard Certificate

All values of the standard certificate are documented in the printout of the Standard Certificate signed by the Quality Assurance Manager.

3.1.4 Add-on for measuring cells for reflection measurement

The USP test can be carried out with 2 different measuring cells (NIRFlex Solids and NIRFlex Fiber Optic Solids). Two add-ons are included to perfectly fit the particular measuring cells, see Fig. 3.2.



① Add-on NIRFlex Fiber Optic Solids

② Standards disk

Fig. 3.2: Scope of delivery

③ Add-on NIRFlex Solids

④ CD containing the Standard Certificate file

3.2 Scope of delivery for solid transmission measurement

There are two different test kits, one for the NIRFlex Solids Transmittance and one for the NIR Checkmaster.

For NIRFlex Solids transmittance N512-008

For NIR Checkmaster: N512-009

Measuring cell NIRFlex Solids Transmittance



Fig. 3.3: USP standard plate for measuring cell NIRFlex Solid Transmittance cell

NIR Checkmaster



Fig. 3.4: USP standard kit for the NIR Checkmaster

3.2.1 USP Standards

The Buchi USP Standards are integrated in a disk. This disk contains 5 neutral density filter standards and 1 standard (Buchi Rare Earth Oxide Standard) for checking the wavenumber accuracy and repeatability. All standards are protected with a window of special glass.

Table 3-2: Technical data of reference material for solid transmission measurement

Standard ID	Standard description	Standard composition
Ref	Empty position, 100% transmissivity	Air
GS 80 %	Neutral density filter with nominal 79.4 % transmissivity	Neutral density filter
GS 64 %	Neutral density filter with nominal 63.1 % transmissivity	Neutral density filter
GS 32 %	Neutral density filter with nominal 31.6 % transmissivity	Neutral density filter
GS 10 %	Neutral density filter with nominal 10 % transmissivity	Neutral density filter
GS 2 %	Neutral density filter with nominal 2 % transmissivity	Neutral density filter
BREOS	Buchi Rare Earth Oxide Standard	Mixture of rare earth oxides

3.2.2 CD containing Standard Certificate file

The Standard Certificate file is delivered in xml-format and contains the expected values (% transmittance and wavenumbers for solid transmission measurement) of the standards in relation to the master used for measurement at the Buchi production site before shipment.

3.2.3 Hardcopy of the Standard Certificate

All values of the standard certificate are documented in the printout of the Standard Certificate signed by the Quality Assurance Manager.

3.3 Software requirements

To carry out the test, the following preconditions must be met:

- The NIRWare Software has to be installed (for a corresponding description, see the NIRWare Software Manual).
- The USP test kit License has to be activated.



IMPORTANT

This action has to be performed by the service engineer only.

- The Standard Certificate file has to be stored on the computer.

NOTE:

We recommend to close all other programs on the computer when running the tests as their execution is quite resource intensive.

The test is performed using the NIRWare Operator Module. It leads the user through the test procedure, performs the calculations according to the USP and generates the protocols.

3.4 Activating the USP test kit license

This procedure may have been performed during the installation of the USP test kit by the service engineer already. If not, proceed as follows:

- Open the NIRWare Management Console.
- Select **Help > Software Registration...**

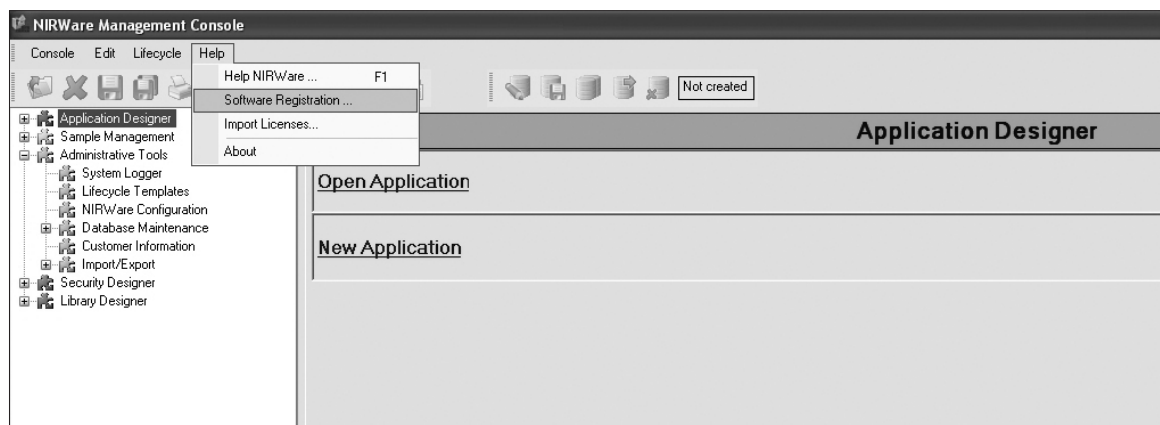
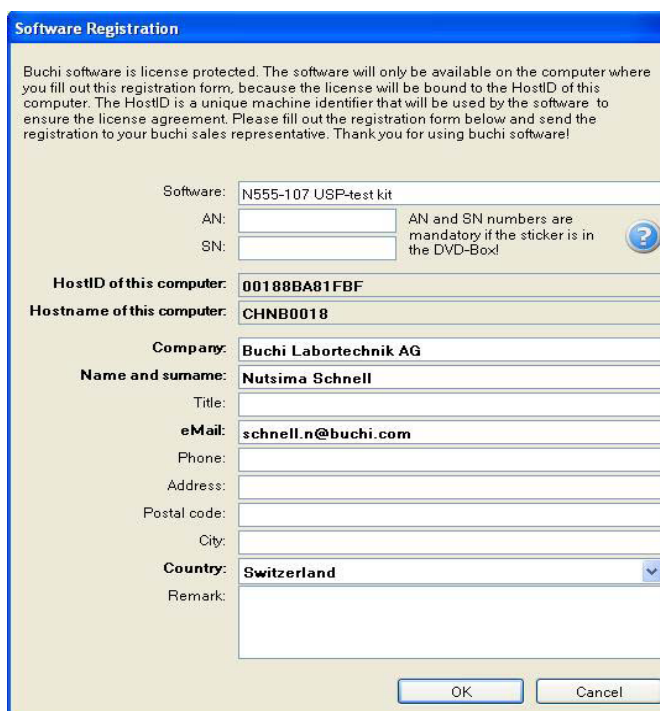


Fig 3.5: Opening the Software Registration in the NIRWare Management Console

- Fill in all necessary information (bold information fields).



Software Registration

Buchi software is license protected. The software will only be available on the computer where you fill out this registration form, because the license will be bound to the HostID of this computer. The HostID is a unique machine identifier that will be used by the software to ensure the license agreement. Please fill out the registration form below and send the registration to your buchi sales representative. Thank you for using buchi software!

Software: N555-107 USP-test kit

AN: AN and SN numbers are mandatory if the sticker is in the DVD-Box!

SN:

HostID of this computer: 00188BA81FBF

Hostname of this computer: CHNB0018

Company: Buchi Labortechnik AG

Name and surname: Nutsima Schnell

Title:

eMail: schnell.n@buchi.com

Phone:

Address:

Postal code:

City:

Country: Switzerland

Remark:

OK Cancel

Fig 3.6: Software Registration

- Confirm with **OK** and save the registration file on your harddisk.
- Send the registration file via e-mail to registration@buchi.com. Please mention in the email that this registration file is for “N555-107 USP-test kit”.

NOTE

Please send the licence request email two days before installation.

By means of this file you can order the license to activate the USP test kit software free of charge. It is possible to order more than 1 license, if necessary. For order details, please contact your local sales representative.

For order details, please contact your local sales representative.

- To import and activate the license, in the NIRWare Management Console select **Help > Import Licenses...** The following window opens:

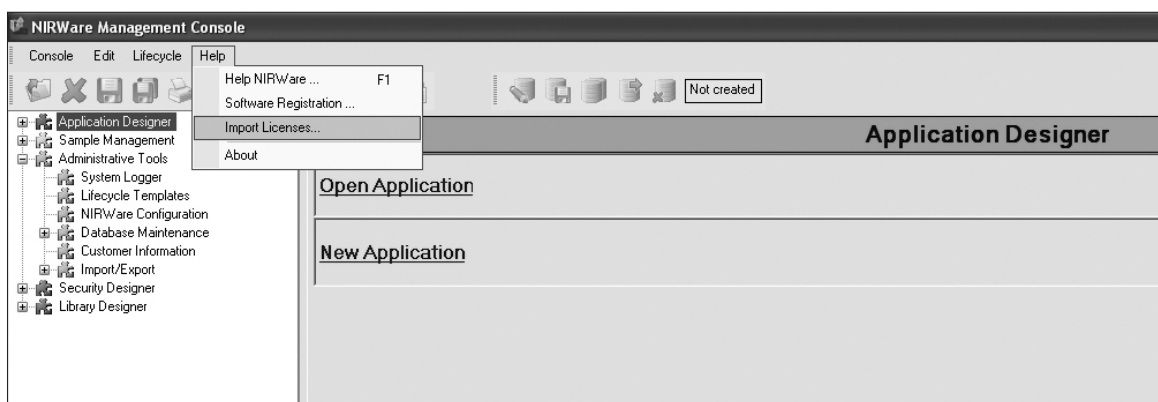


Fig 3.7: Importing and activating the license

- Now search the location of the license file and import.

NOTE

The user needs administrator rights for the operating system to import the license file.

4 Description of the standards and the test

This chapter explains the function of the UPS Performance Verification Kit and the corresponding Wavelength Standards.

4.1 Introduction

The Buchi USP Performance Verification Kit is an option designed for the system validation of the NIRFlex N-500 FT-NIR spectrometer in the pharmaceutical industrial environment.

The USP (United States Pharmacopeia) has published criteria for the validation of an NIR spectrometer used in the pharmaceutical industry (First supplement to USP 35-NF 30, General Information Chapter <1119>). The European Pharmacopeia has published corresponding criteria (7th Edition, Chapter 2.2.40).

This means that users of NIR spectrometers in this industry need to have appropriate standards for testing and, for their convenience, a software that leads the operator through the test and automatically generates the required protocols.

4.2 Photometric linearity test

The system linearity test checks if the response of the spectrometer correlates linearly with a linear signal attenuation.

Gray standards for reflection measurement and neutral density filters for solid transmission measurement have a “nominal” value measured under a certain optical arrangement. In most cases only under this special optical arrangement the response will match the nominal value. For all other systems the real response has to be measured individually.

According to the USP, a set of gray standards (for reflection measurement) and a set of neutral density filters have to be measured once to get initial values. After a certain time period the test has to be repeated and the deviation to the first measurement must not exceed the acceptance criteria.

For this USP test protocol, the five different percentages of gray standards (5, 10, 20, 40 and 80%) for reflection measurement and the five different percentages of neutral density filters (approximately 2, 10, 31.6, 63.1 and 79.7 %) for solid transmission measurement are measured. Three different wave-numbers are evaluated (5000, 6250 and 8330 cm^{-1} for reflection measurement and 6250, 8330 and 10000 cm^{-1} for solid transmission measurement). The tolerance of the intercept is ± 0.05 , the tolerance of the slope is ± 0.05 .

4.3 Noise test

The noise test typical involves measurements of reference materials at high and low flux. The high flux noise is measured at approximately 100 % signal level of the instrument using a 99 % reflective white standard for reflection measurement and a 100 % transmission (empty position), the low flux noise is measured with an approximately 10 % gray standard reflectance material for reflection measurement and a neutral density filter with nominal 10 % transmissivity for transmission measurement.

The spectrum is split into sections of 300 cm^{-1} each. The peak-to-peak noise is measured for each section and used for the calculation of the mean noise and the maximum noise at both high flux and low flux. The upper limit of the mean value of high flux is 0.3×10^{-3} and the maximum value is 0.8×10^{-3} . The upper limit of the mean value of low flux is 1.0×10^{-3} and the maximum value is 2.0×10^{-3} .

4.4 Wavenumber accuracy

The wavenumber accuracy of the system is checked with a standard material which has well defined peaks in the NIR range. BREOS (mixtures of rare earth oxides) is used for demonstrating wavelength dispersion. The wavenumber accuracy test is evaluated at the 4 peaks over the range. The tolerance is documented in the test protocol.

4.5 Wavenumber repeatability

The European Pharmacopoeia requests the test of the wavenumber repeatability. The test is performed using the same rare earth oxide standard. The standard deviation of the 4 peaks over the range for 3 repeated measurements should be within ± 0.20 .

5 Overview over the steps to be carried out

This chapter gives a short overview over all steps to be carried out to prepare, execute and finish the USP Performance Verification Test in chronological order.

To carry out the test, proceed as follows:

- Make sure that the NIRWare Software is installed and that the USPKit License (N555-107) is activated.
- Install the measuring cell you are operating with (NIRFlex Solids or NIRFlex Fiber Optic Solids for reflection measurement, NIRFlex Solids Transmittance or the NIR Checkmaster for solid transmission measurement) to the instrument. If the customer has more than one measuring cell, the installation procedure has to be performed for each cell individually.
- Switch on the computer.
- Switch on the instrument and let it warm up for at least 1 hour.
- Establish the connection between the computer and the instrument, start the test and follow the instructions on the screen.
- When the test is successfully finished, print the USP Protocol, sign it and file the print-outs according to your SOP.

NOTE

The USP regular test can be performed after the USP test kit has been installed successfully.

The installation of a USP test kit has to be performed by an authorized service engineer.

For information on the installation procedure, please refer to the Installation Manual of the USP Performance Verification Kit.

6 SOP for performing the USP regular test

This chapter gives instructions on how to carry out the USP Performance Verification Test properly and safely.

6.1 Preparation

To prepare the test, proceed as follows:

- Place the measuring cell on the instrument.
- Switch on the instrument and let it warm up for at least 1 hour.
- Attach the proper USP Standard add-on to the measuring cell.
- In case the ControlSystem Service is not started automatically, click **Start > Programs > Buchi > NIRsolutions > Tools > ControlSystem Service Manager**.
- Double click the ControlSystem Service Icon.



Fig. 6.1: ControlSystem Service Icon

The following dialog opens:



Fig. 6.2: ControlSystem Service Manager

- Click the green start button. The ControlSystem Service starts.
- Start the NIRWare Operator Module and log in:



Fig. 6.3: Login dialog

- Click the Service icon . The menu structure on the left is now available.

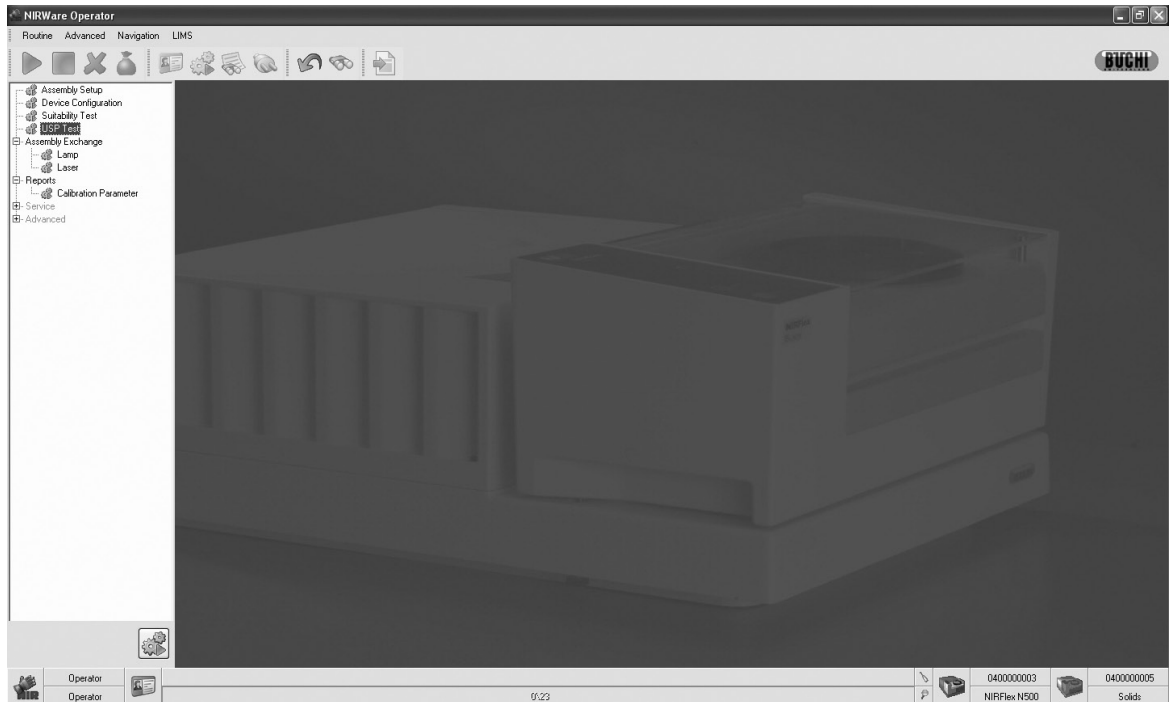


Fig. 6.4: NIRWare Operator

- Double-click **USP Test**. The dialog **Regular USP Test** opens.

6.2 Working with several USP kits in one database

If you have several USP kits (different serial numbers) with the same type of USP kit (reflection or solid transmission measurement), the window below is displayed.

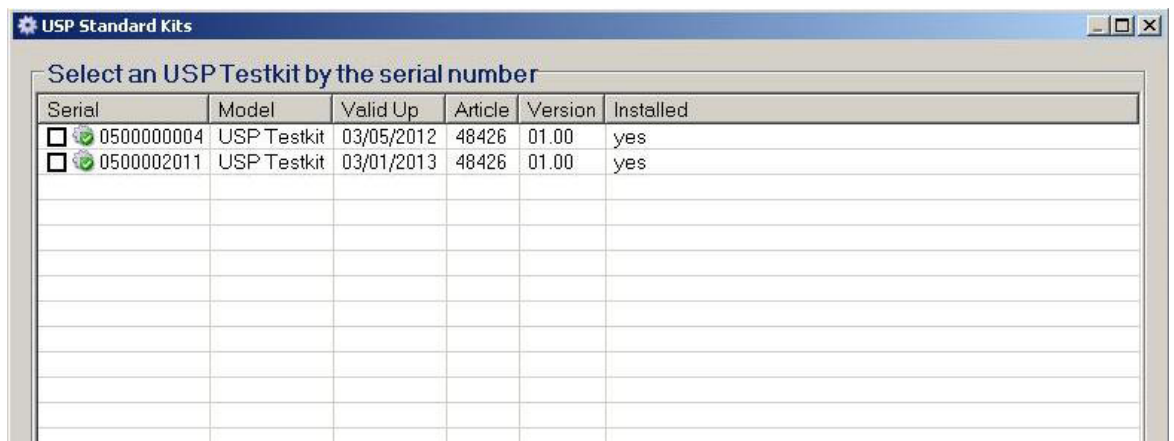


Fig. 6.5: Activating the serial number of the desired USP kit in case several USP kits of the same type are installed in one database

Activate the corresponding checkbox of the test kit that is carried out and click the green arrow to confirm.

If you carry out a different type of USP kit (reflection and solid transmission measurement) in one database, the installed measuring cell will automatically recognize the software.



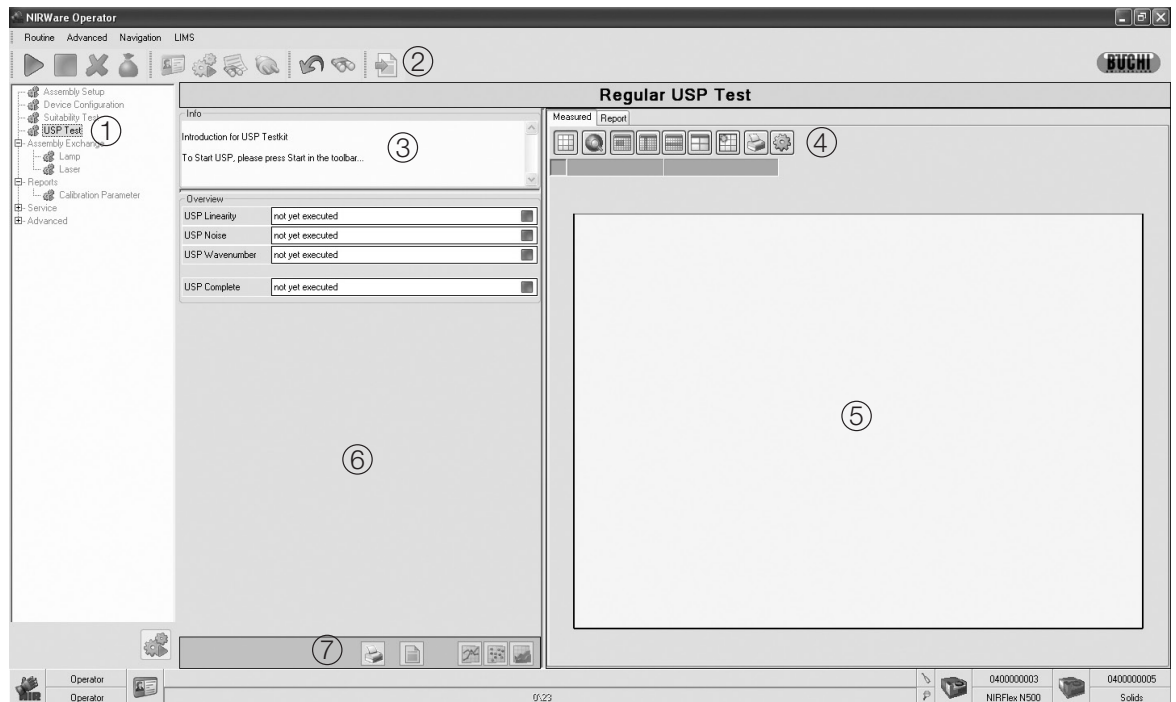
ATTENTION

If you want to carry out a USP regular test for several measuring cells, after each USP regular test

leave the service dialog by clicking the Back icon , change the measuring cell and then open the service dialog again.

6.3 Description of the main window in the NIRWare Operator

The main window, where you navigate during the test procedure consists of several areas:



① Menu structure service menu

② Toolbar

③ Info area

④ Iconbar for graphics

Fig. 6.6: Description of the main window

⑤ Fields for axis position

⑥ Graphic and report area

⑦ Test value area

6.3.1 Menu structure service menu

The USP Test menu can be started from here.

6.3.2 Toolbar

The only icons needed from the toolbar to carry out the test are the start and the stop icon.



Click this icon to start the measurement.



Click this icon to stop the measurement.

6.3.3 Info area

In the info area the steps to be carried out are displayed. By means of this information you are guided through the test procedure step by step.

When a test was started, the steps currently carried out are listed. When an error occurs, a corresponding error description is given here.

6.3.4 Iconbar for graphic and report





Icons for graphic and report operations are available here.

6.3.5 Graphic and report area

A preview of selected graphics and reports is shown here.

6.3.6 Test value area - Overview

The test value area gives an overview over the tests to be executed and an indication whether they were passed or not (in case they have been executed, yet).

Overview	
USP Linearity	not yet executed 
USP Noise	not yet executed 
USP Wavenumber	not yet executed 
USP Complete	not yet executed 

6.3.7 Iconbar for test values and spectra

After the tests are finished the test values of the individual tests can be displayed by means of the iconbar for test values and spectra.



Overview Icon: Click here to get an overview over all test results and all measured spectra.



Linearity Icon: Click here to display the test values of the linearity test in the test value area and the corresponding spectra in the graphic and report area.



Noise Icon: Click here to display the test values of the noise test in the test value area and the corresponding spectra in the graphic and report area.



Wavelength Icon: Click here to display the test values of the wavenumber accuracy test in the test value area and the corresponding spectra in the graphic and report area.

6.3.8 Iconbar for graphics

The iconbar for graphics enables to modify the display of the test data (spectra) when the test was carried out.



Grid On/Off



Zoom to overview



Zoom both axis



Zoom horizontally



Cross cursor On/Off



Nearest Point On/Off



Print: Print the graphic on the connected printer. When this icon is clicked a print preview appears in which different print settings can be defined.



Edit chart. When this icon is clicked a chart editor appears, in which different settings concerning graphic display can be defined.

6.4 Positioning the standards

In order to get reproducible results it is essential to position the samples in a highly reproducible way. For this purpose, two add-ons are available for reflection measurement, depending on whether you carry out the measurement with the NIRFlex Solids or with the NIRFlex Fiber Optic Solids. For solid transmission measurement with the NIRFlex Solids Transmittance the standard plate is used, for solid transmission measurement with the NIR Checkmaster the standard disk is used.

6.4.1 NIRFlex Solids

- Place the Standards add-on on the NIRFlex Solids measuring cell, so that the round bump on the measuring cell fits into the large round notch on the add-on and the small pin catches.
- Insert the standards disk with the standard windows face down into the add-on, so that the two facing pins at the bottom of the standards disk fit into the provided slots in the add-on.



① Measuring cell NIRFlex Solids
② Standards add-on

③ Standards disk

Fig. 6.7: Standards add-on for measuring cell NIRFlex Solids

6.4.2 NIRFlex Fiber Optic Solids

Mount the Standards add-on NIRFlex Fiber Optic Solids onto the tip of the fiber optic probe as follows:

- Undo the top screw and remove the spring.
- Insert the standards disk into the adapter, so that the standards are facing the optical window of the probe.
- Put the spring back and refix the screw, see Fig. 6.8.

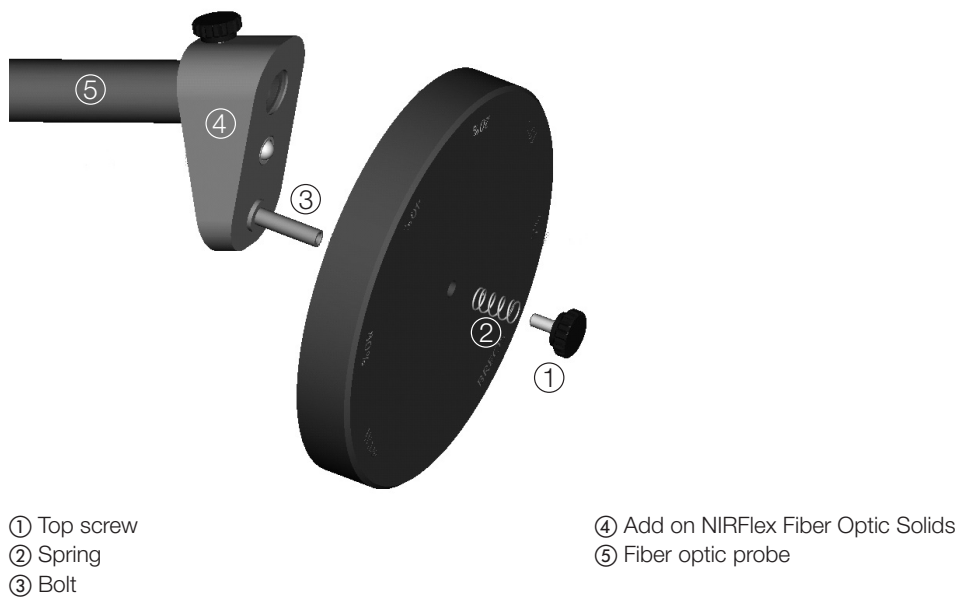


Fig. 6.8: Mounting the standards disk to the add-on NIRFlex Fiber Optic Solids

6.4.3 NIRFlex Solids Transmittance

- Place the standard plate on the NIRFlex Solids Transmittance measuring cell, see Fig. 6.9.

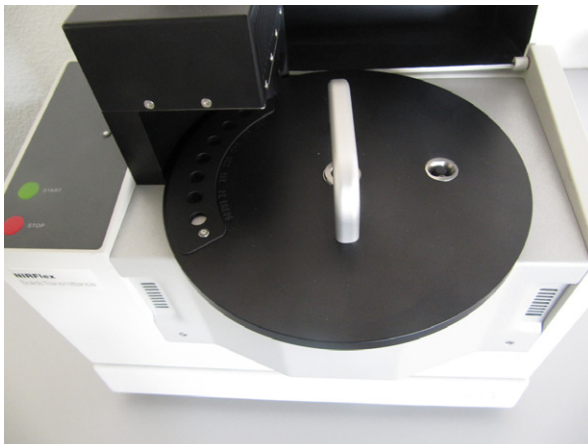


Fig. 6.9: Standard plate on NIRFlex Solids Transmittance

6.4.4 NIR Checkmaster


- Insert the USP standard disk into the NIR Checkmaster, see Fig. 6.10.



Fig. 6.10: Standard kit on the NIR Checkmaster

6.5 Starting the test

6.5.1 Reflection measurement

- To start the test, click the start icon. 

For the measuring cell NIRFlex Solids the test is automatically carried out.
For the measuring cell NIRFlex Fiber Optic Solids you will be asked to mount the standard disk to the NIRFlex Fiber Optic Solids tip.

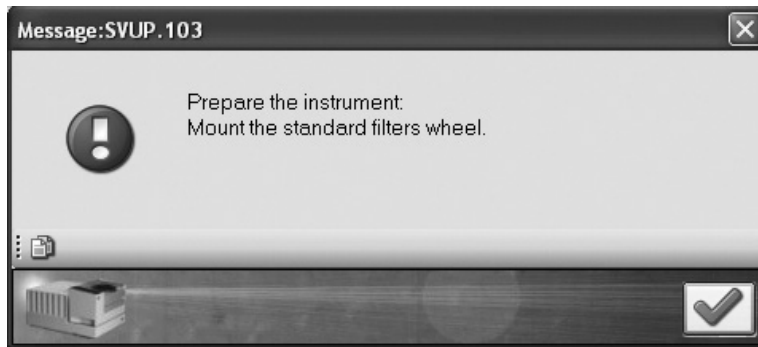


Fig. 6.11: Message box for preparing the test with the measuring cell NIRFlexFiber Optic Solids

After mounting and clicking the green arrow to accept the message, you will be guided through the test procedure. Follow the instructions on the screen, the messages will tell you which standard position has to be placed in front of the NIRFlex Fiber Optic Solids tip:

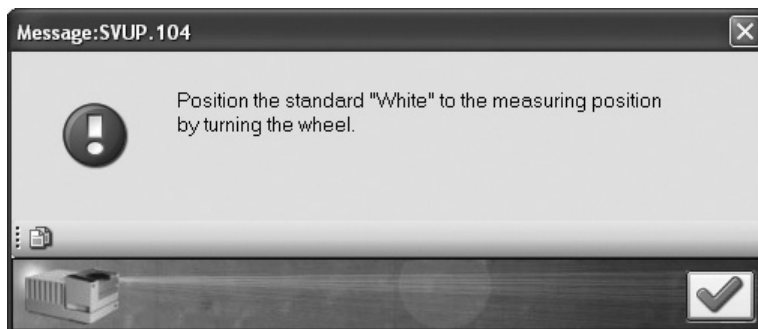


Fig. 6.12: Message box for running the test with the measuring cell NIRFlex Fiber Optic Solids

To change the position, turn the standards disk.

NOTE

The standard IDs are written on the back of the standards disk.

The current steps are indicated in the info area. The corresponding progress can be seen from the progress bar below.

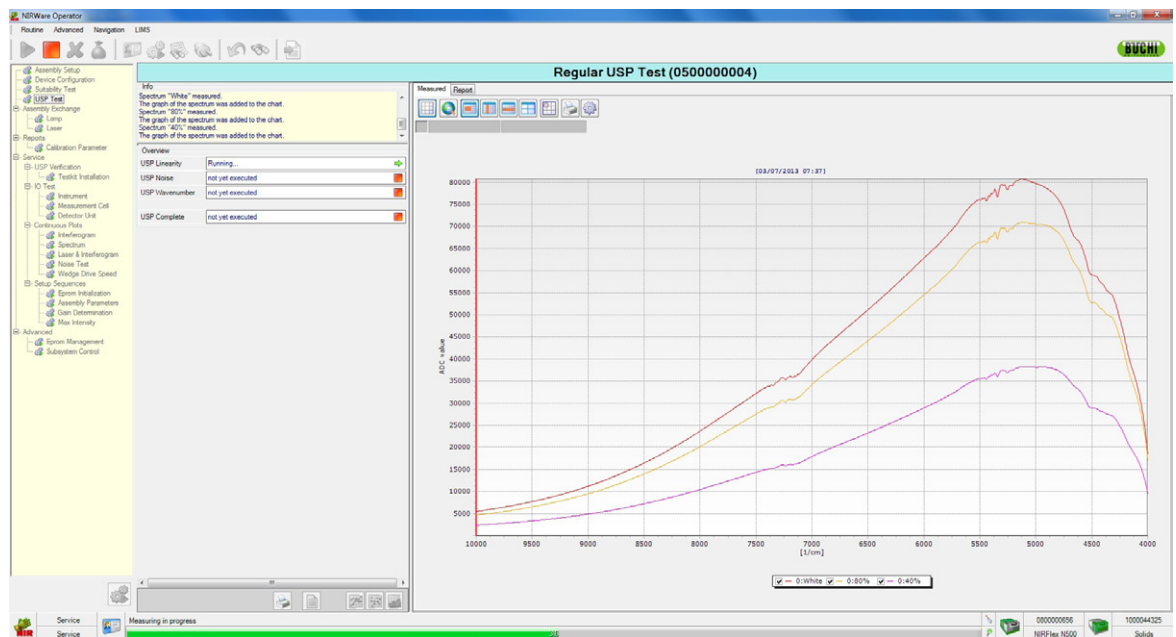


Fig 6.13: Measurement in progress

6.5.2 Solid Transmission measurement

For the measuring cell NIRFlex Solids Transmittance the test is automatically carried out.

For the NIR Checkmaster the following message boxes appear. Confirm them by clicking the green arrow.



Fig. 6.14: Message box

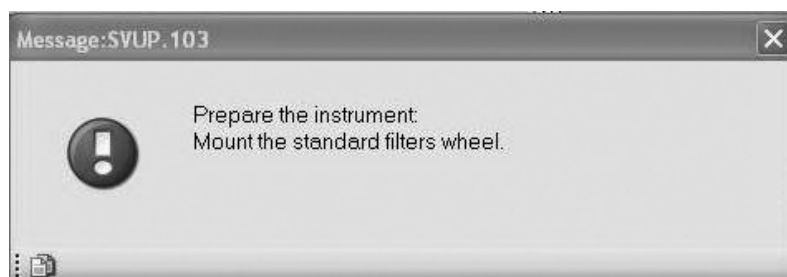


Fig. 6.15: Message box

After mounting the standard kit and clicking the green arrow to confirm the message, you will be guided through the test procedure. Follow the instructions on the screen, the messages will tell you which standard position has to be placed in front of the measuring position:

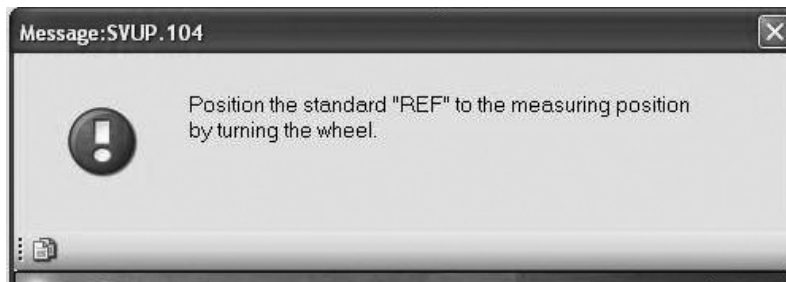


Fig. 6.16: Message box for running the test with the NIR Checkmaster

To change the position, turn the standards disk. The measuring position is marked, see also Fig. 6.17.



Fig. 6.17: Measuring position marked on the standards disk, an example of BREOS measuring position

NOTE

The standard IDs are written on the back of the standards disk. If the standard disk is installed on the NIR Checkmaster the standard IDs on the standards disk are difficult to see. To be able to measure the right standard, the IDs are also written on the edge of the disk. The ID written above the position where the two slots overlap indicates the standard which is currently in measuring position (see also Fig. 6.17 above).

When the tests are finished you are prompted to authorize, i.e. to enter your user name and password in the login dialog again.

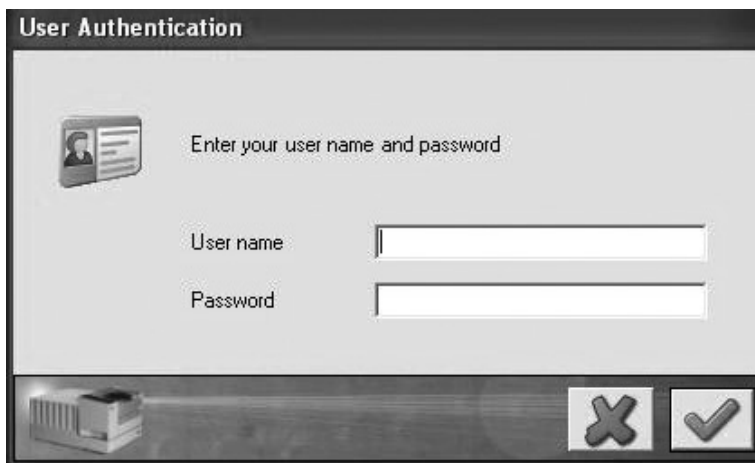


Fig. 6.18: Login dialog for authorization

Click the green check mark to confirm.

You now have an overview over the test results, the iconbar for test values and spectra is active and the measured spectra are displayed in the graphic and report area.

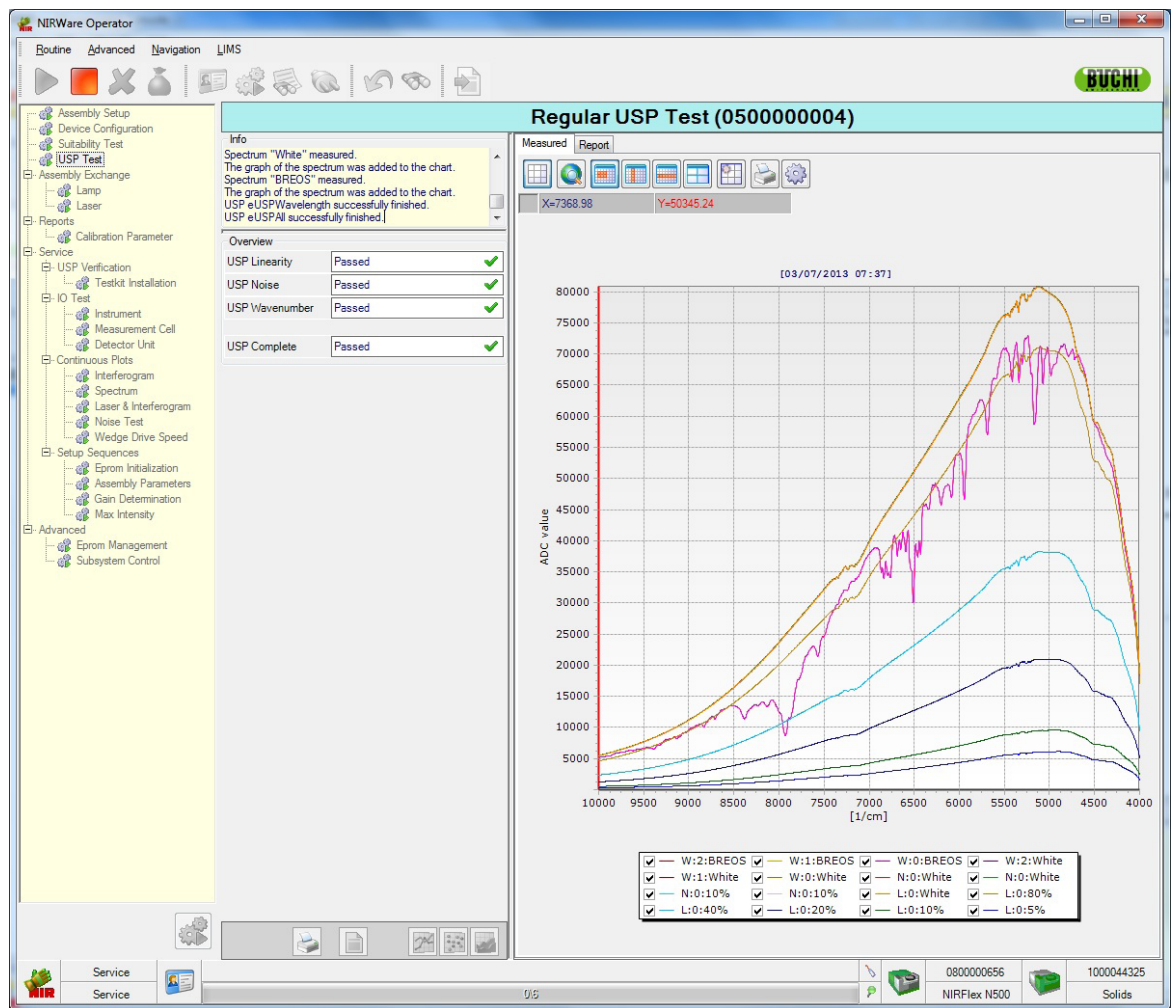


Fig. 6.19: Overview over the measuring results (for reflection measurement)

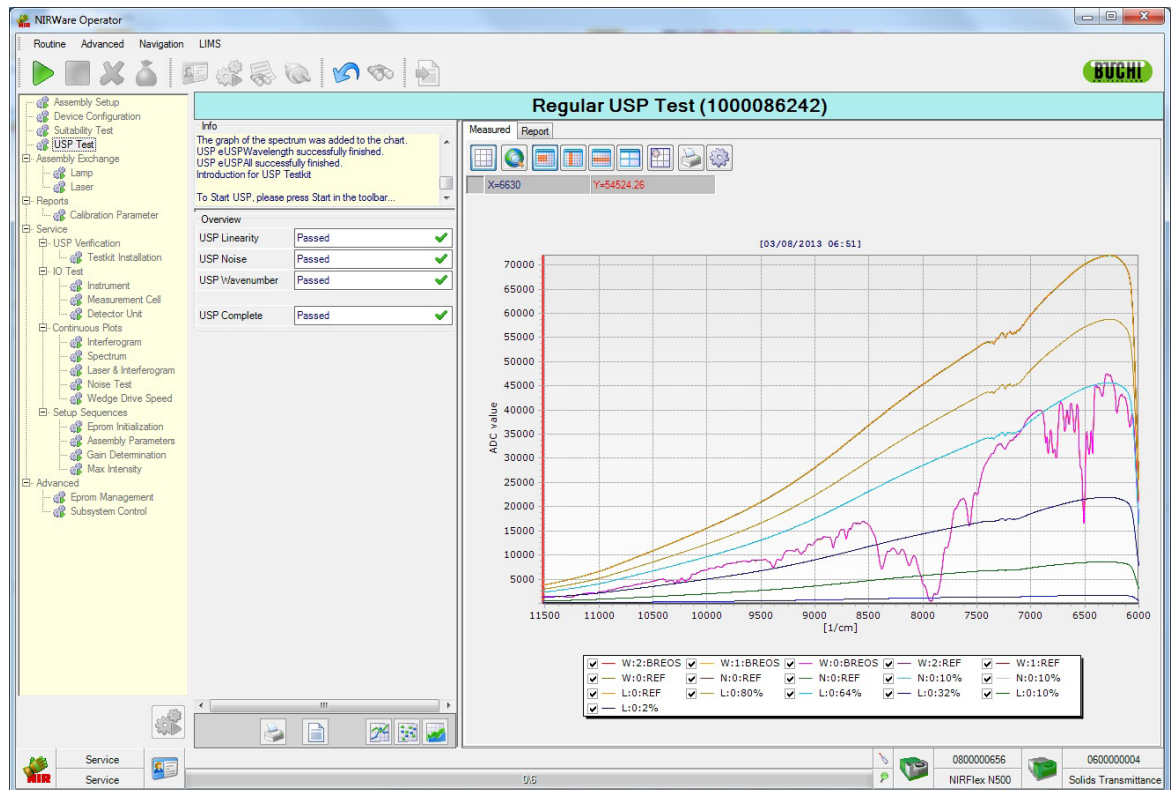


Fig. 6.20: Overview over the measuring result (for solid transmittance measurement).

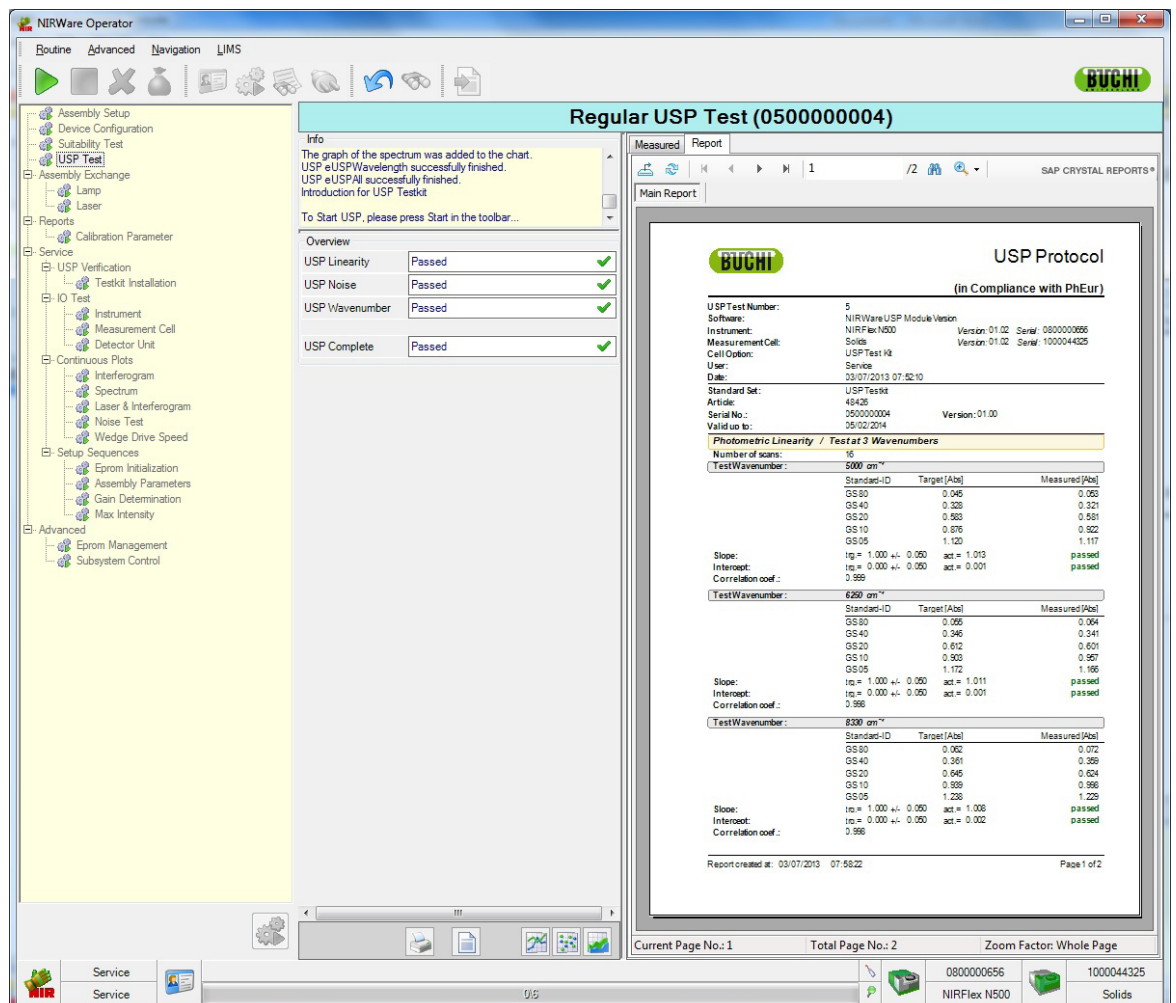


Fig. 6.21: Test report

The USP regular test is passed when the measured values are within the tolerance limits of the target values. The target values result from the target calculation values (Trg calc.) in the USP installation.

In case the test fails, you will get a corresponding message (see example below). In this case, please check whether the USP test kit is correctly installed and clean and do corrective action. If a repeated test still fails, please contact a Buchi service engineer.



Fig. 6.22: Message USP test failed as example

6.6 Evaluating the tests

6.6.1 Linearity test

To see the results of the linearity test, click the Linearity Icon .

Measured

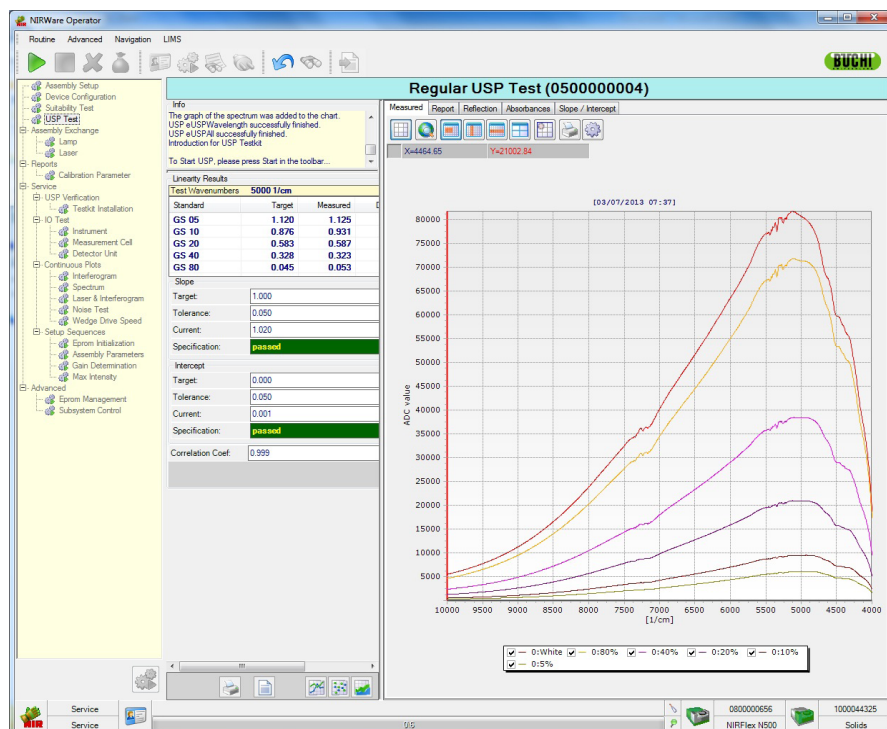


Fig. 6.23: Raw spectra of the linearity test for USP reflection measurement kit

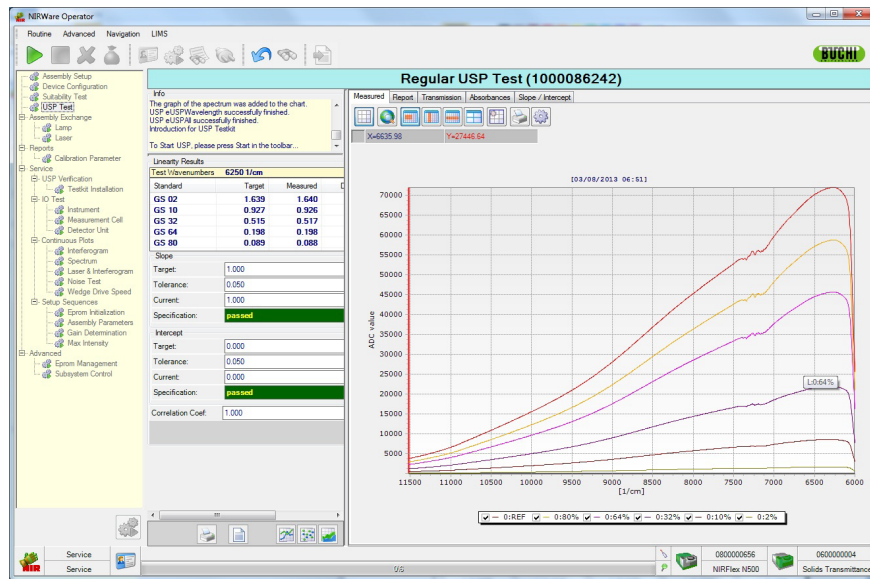


Fig. 6.24: Raw spectra of the linearity test for USP transmission measurement kit

NOTE

For the USP solid transmission measurement kit the raw spectra of the linearity test are different (different standards and wavelength range).

On the **Measured** tab the measured raw spectra are displayed in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed.

You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated.

Via the iconbar the spectra can be edited, printed and displayed in different views.

Report

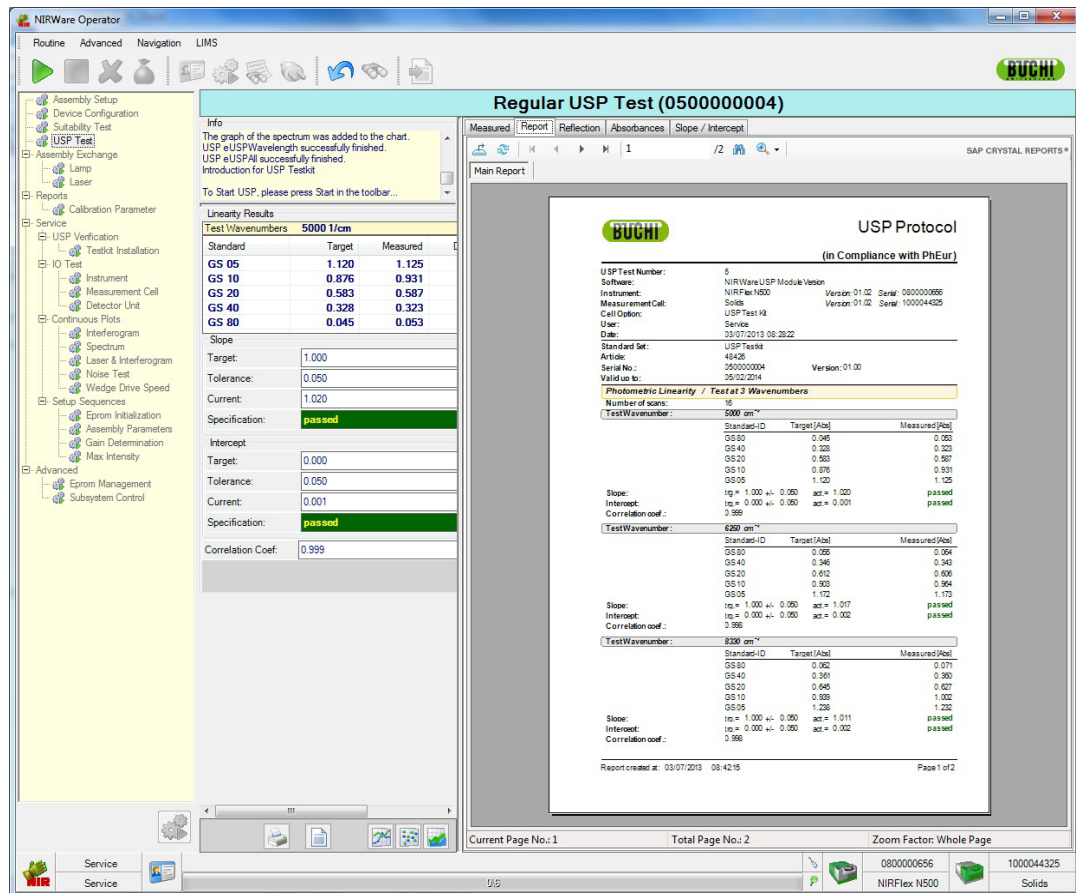


Fig. 6.25: USP Protocol for USP reflection measurement kit

NOTE

For the solid USP transmission measurement kit the details are different (different test wavenumbers and standards).

On the **Report** tab the USP Protocol automatically created when the tests are finished is displayed. There are some additional functions available via the report iconbar:

Use the icons in the iconbar to navigate within the report and to process it:



Export Report Icon: Click here to export the report.



Refresh Icon: Click here to refresh the report and to get back to the default display.



Go to first page icon.



Go to previous page icon.



Go to next page icon.



Go to last page icon.



Search Text Icon: Click here to search for text within the report. A dialog opens, where you can enter the text to be searched for. After that click **Find Next**.



Zoom Icon: Click the arrow. A drop-down menu opens, where you can select the size in which the report is displayed by clicking on the corresponding percentage.

Reflection

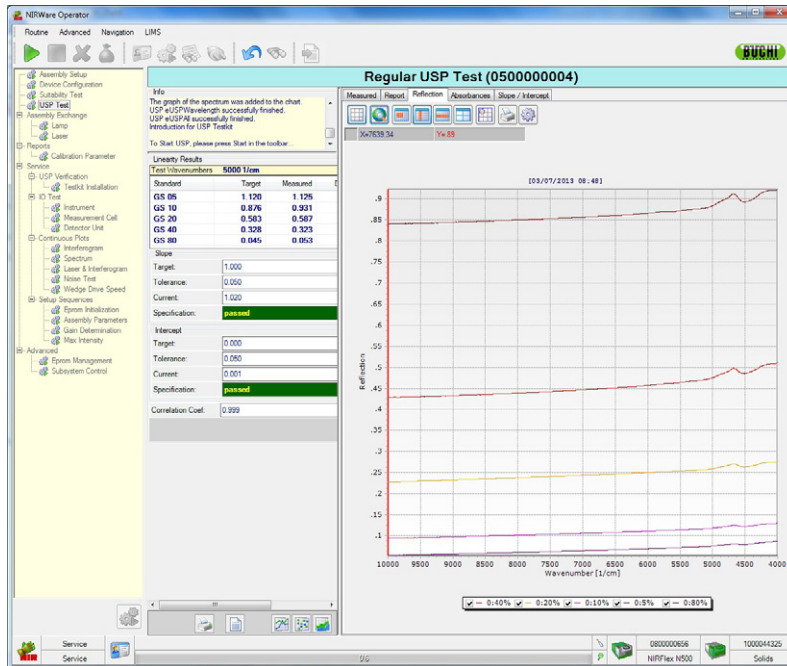


Fig. 6.26: Reflection spectra of the linearity test for the USP reflection measurement kit

Transmission

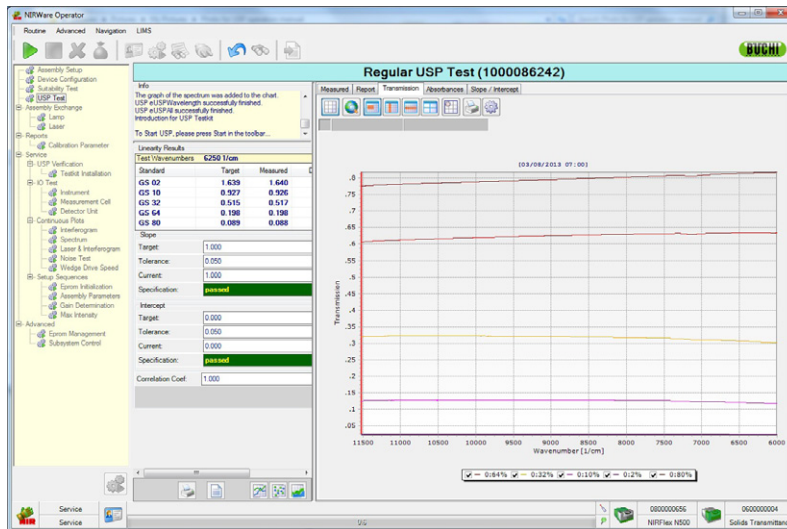


Fig. 6.27: Transmission spectra of the linearity test with the USP solid transmission measurement kit

For the reflection measurement the measured reflection spectra are displayed in different colors on the **Reflection** tab and for the solid transmission measurement the measured transmission spectra are displayed in different colors on the **Transmission** tab. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed. You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated. Via the iconbar the spectra can be edited, printed and displayed in different views.

Absorbances

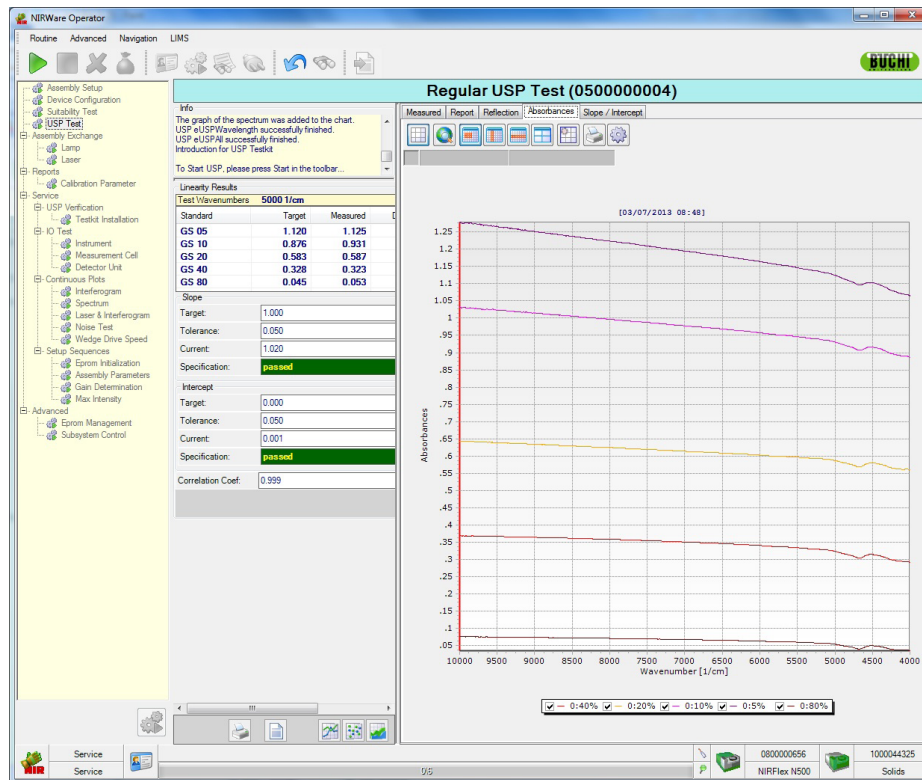


Fig. 6.28: Absorbance spectra of the linearity test for the USP reflection measurement kit

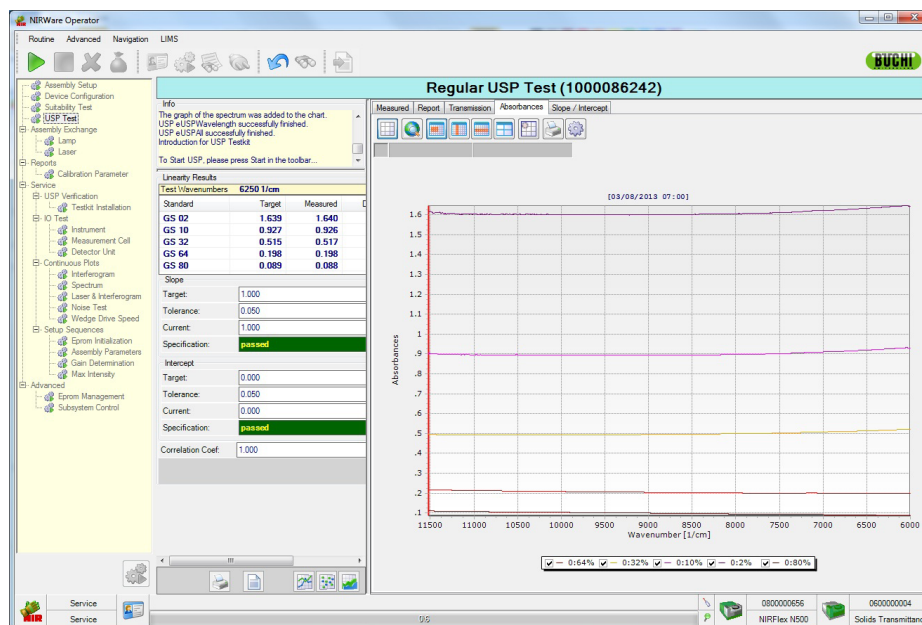


Fig. 6.29: Absorbance spectra of the linearity test for the USP solid transmission measurement kit

On the **Absorbances** tab the measured absorbance spectra are displayed in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed. You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated. Via the iconbar the spectra can be edited, printed and displayed in different views.

Slope/Intercept

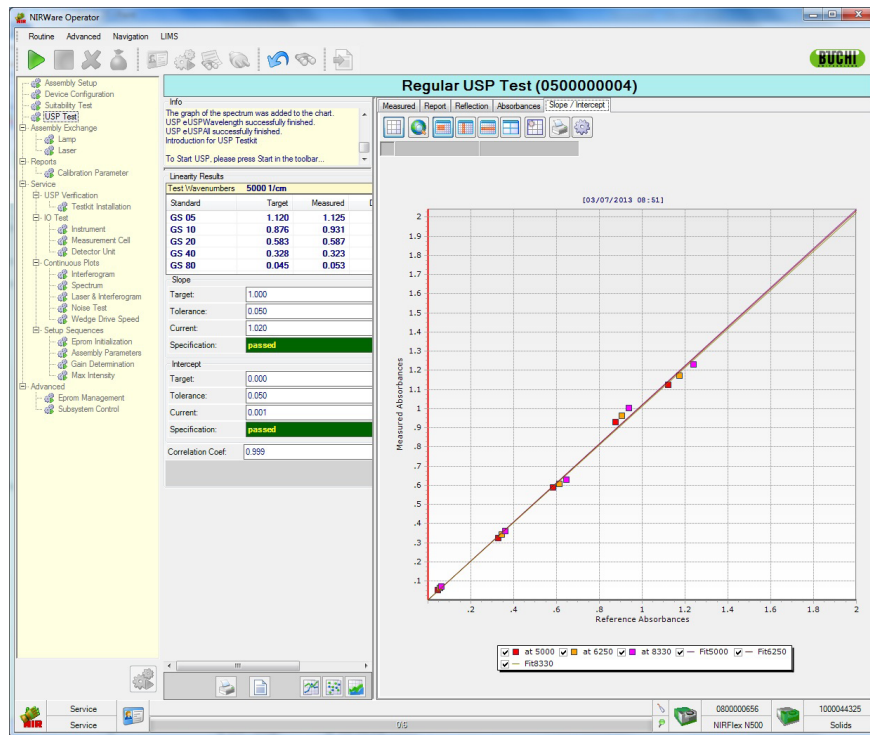


Fig. 6.30: Slope/Intercept of the linearity test for the USP reflection measurement kit

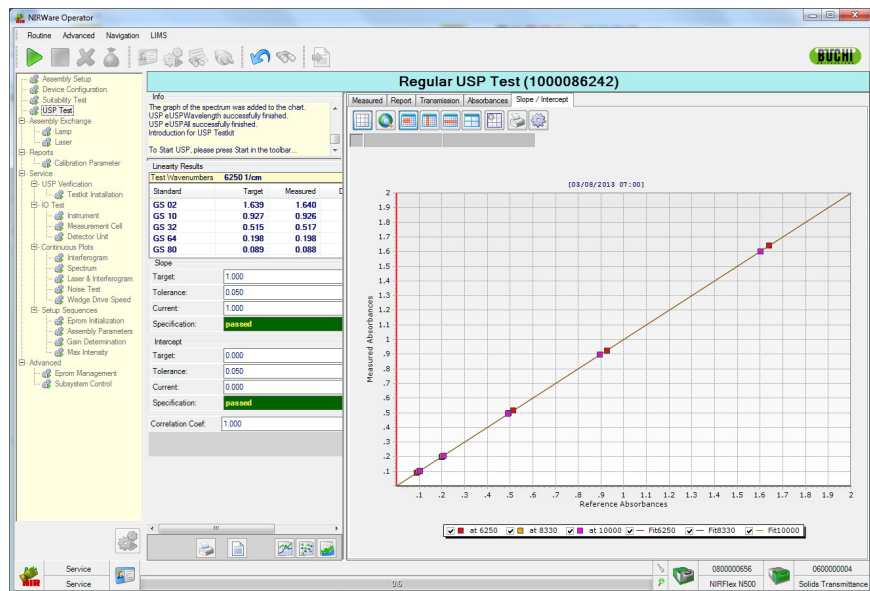


Fig. 6.31: Slope/Intercept of the linearity test for the USP solid transmission measurement kit

On the **Slope/Intercept** tab the corrected reference absorbances are displayed in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed. You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated. Via the iconbar the spectra can be edited, printed and displayed in different views.

6.6.2 Noise test



To see the results of the noise test, click the Noise Icon.

Measured

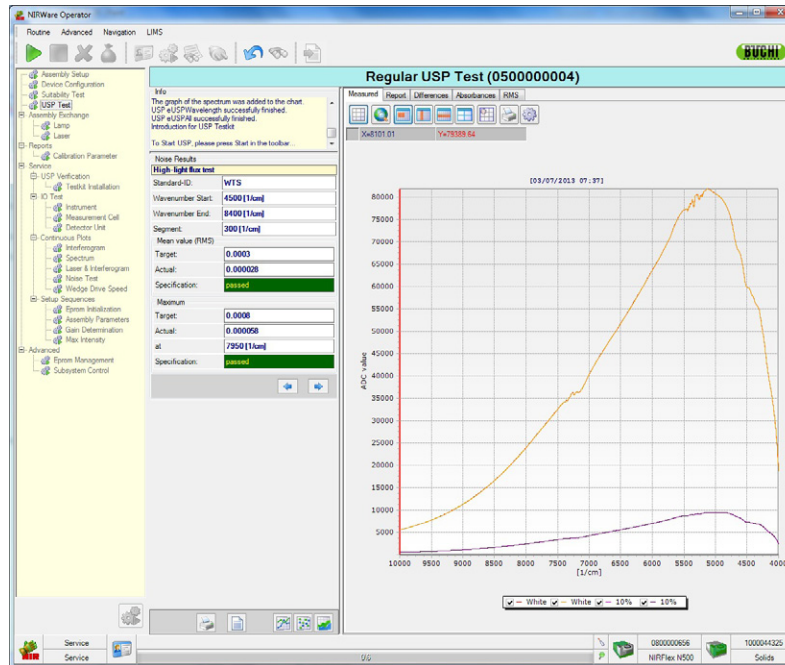


Fig. 6.32: Raw spectra of the noise test for USP reflection measurement kit

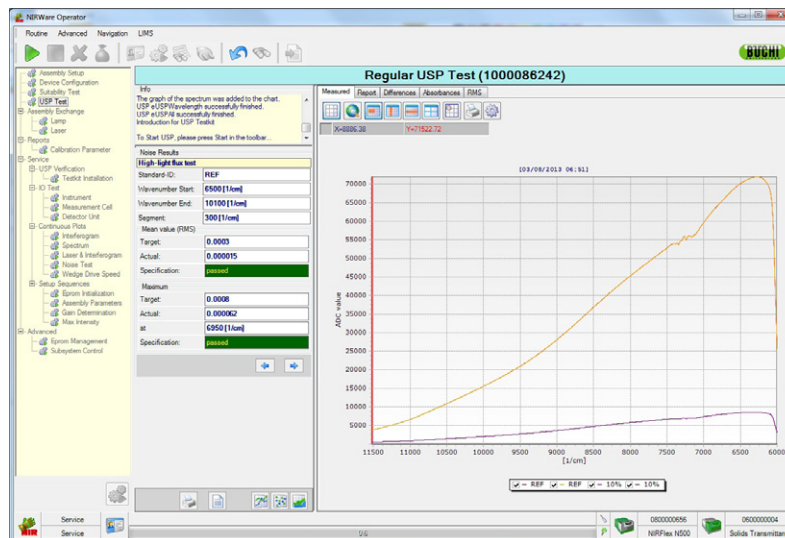


Fig. 6.33: Raw spectra of the linearity test for USP solid transmission measurement kit

On the **Measured** tab the raw spectra of the two measured standards are displayed in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed. You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated.

Via the iconbar the spectra can be edited, printed and displayed in different views.

Report

On the report tab, the USP protocol is shown. The details are the same as in the report tab explained on page 29.

Differences

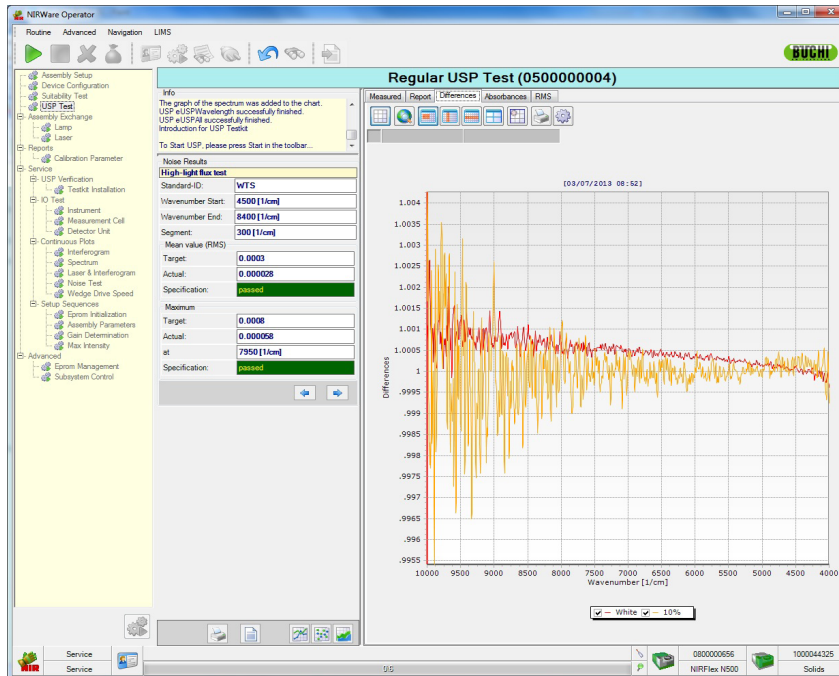


Fig. 6.34: Noise spectra of the noise test for USP reflection measurement kit

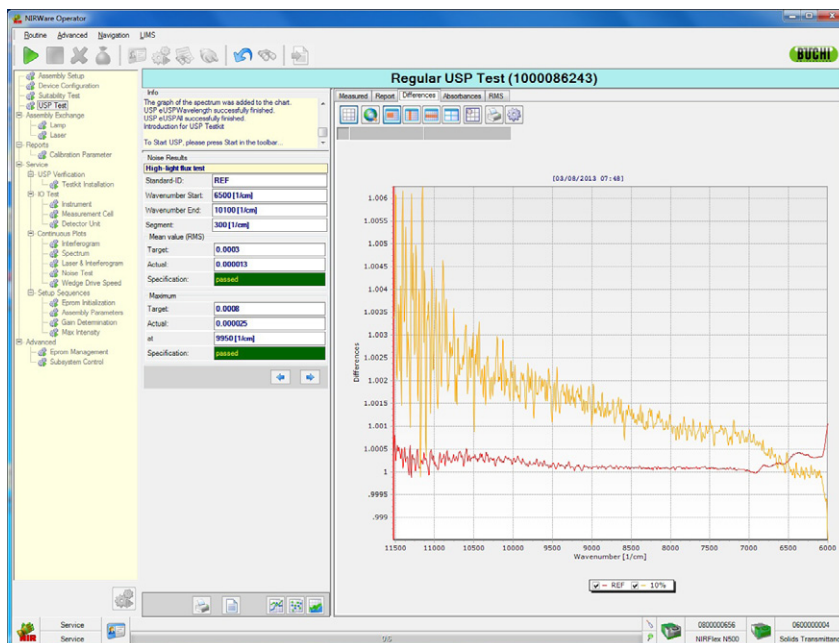


Fig. 6.35: Noise spectra of the noise test for USP solid transmission measurement kit

On the **Differences** tab the noise spectra of the two measured standards are displayed in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed. You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated. Via the iconbar the spectra can be edited, printed and displayed in different views.

Absorbances

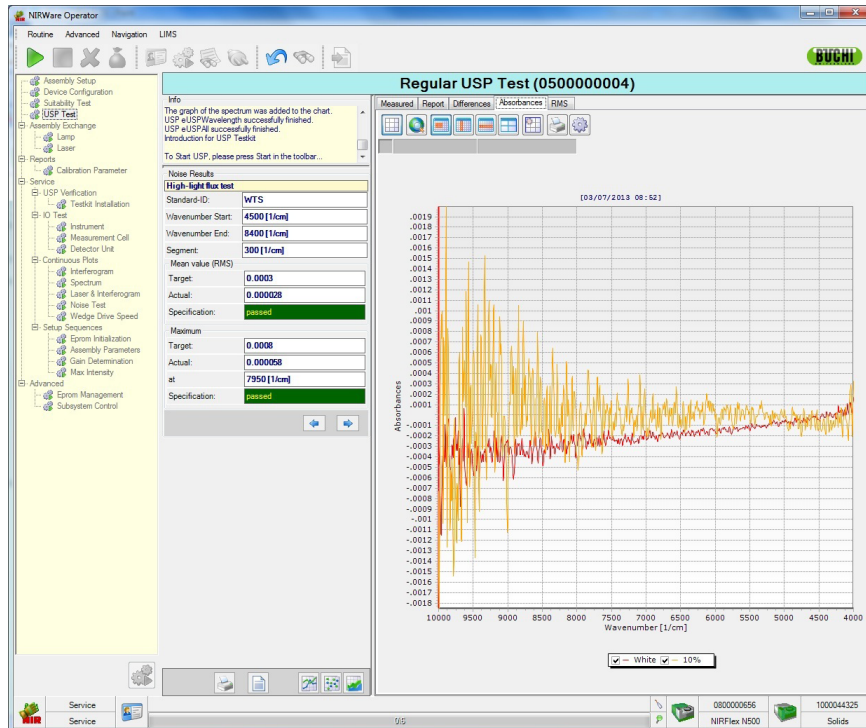


Fig. 6.36: Absorbance spectra of the noise test for USP reflection measurement kit

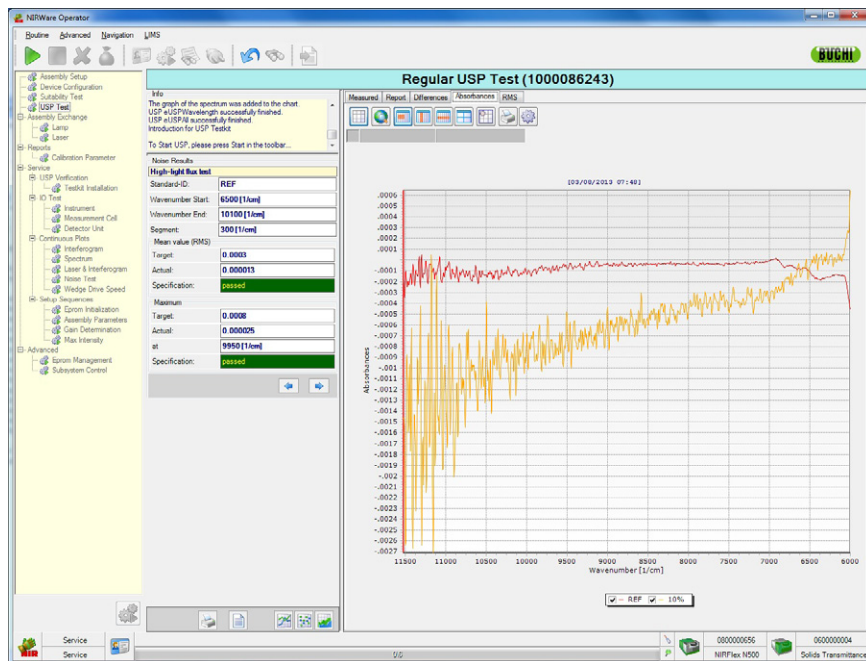


Fig. 6.37: Absorbance spectra of the noise test for USP solid transmission measurement kit

On the **Absorbances** tab the absorption spectra of the two measured standards are displayed in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed.

You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated.

Via the iconbar the spectra can be edited, printed and displayed in different views.

RMS

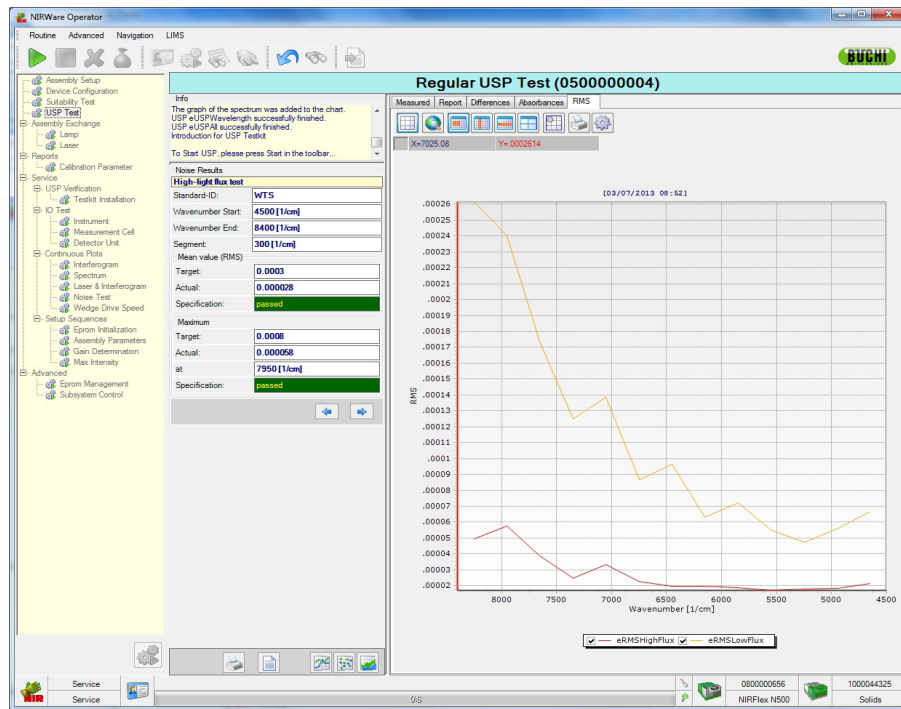


Fig. 6.38: Root Mean Squares spectra of the noise test for USP reflection measurement kit

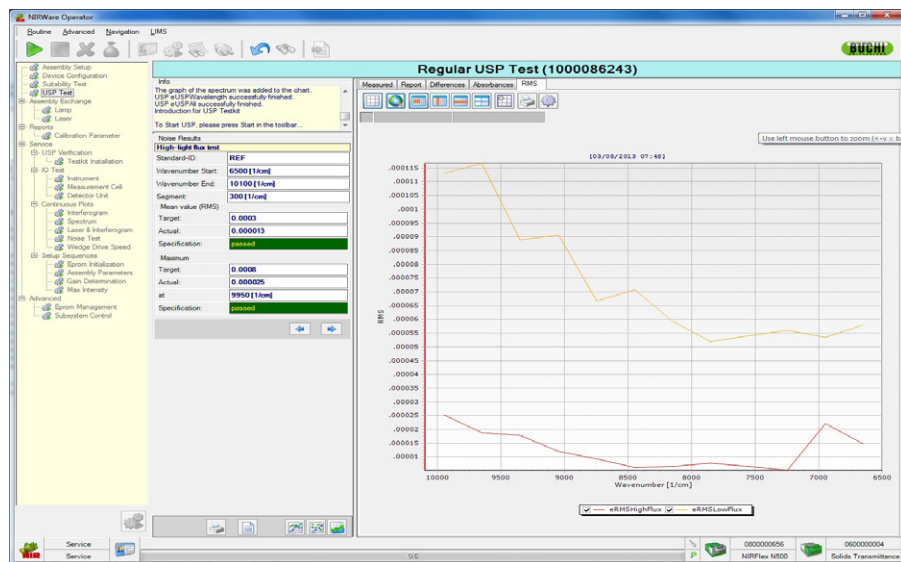


Fig. 6.39: Root Mean Squares spectra of the noise test for USP solid transmission measurement kit

On the **RMS** tab the Root Mean Squares spectra of the two measured standards are displayed in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed. You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated.

Via the iconbar the spectra can be edited, printed and displayed in different views.

6.6.3 Wavelength test

To see the results of the wavelength accuracy and repeatability test, click the Wavelength Icon.



Measured

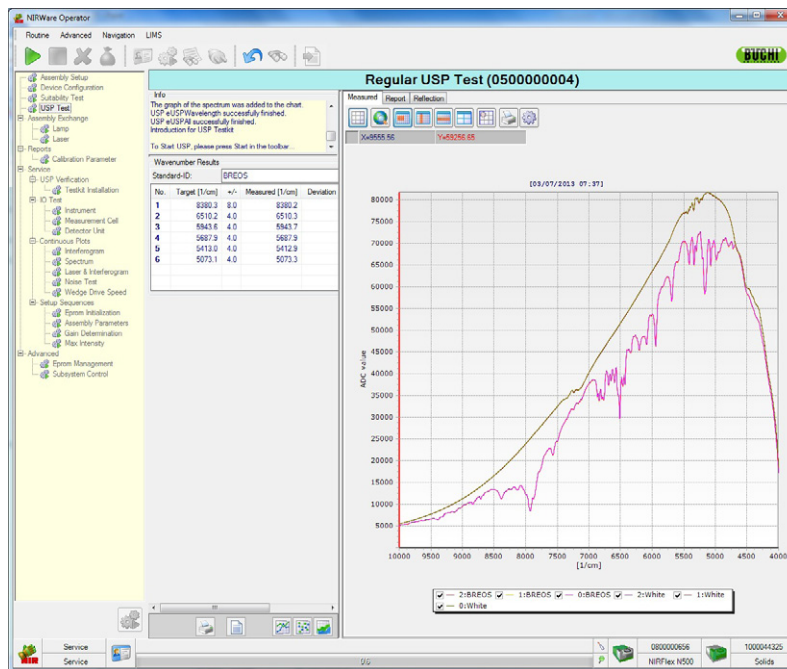


Fig. 6.40: Raw spectra of the wavelength accuracy test for USP reflection measurement kit

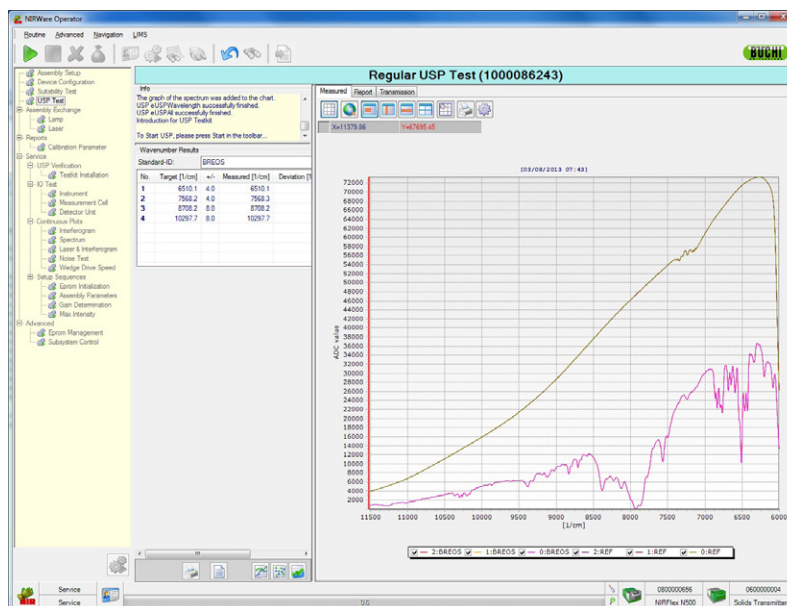


Fig. 6.41 Raw spectra of the wavelength accuracy test for USP solid transmission measurement kit

On the **Measured** tab the measured raw spectra are displayed in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed.

You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated.

Via the iconbar the spectra can be edited, printed and displayed in different views.

Report

On the report tab, the USP protocol is shown. The details are the same as in the report tab explained on page 29.

Reflection

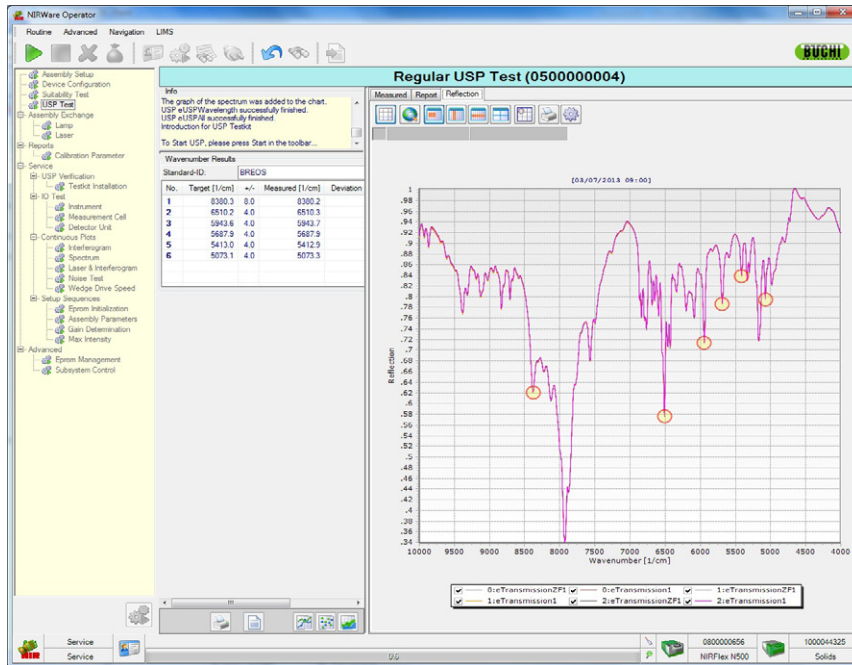


Fig. 6.42: Reflection spectra of the wavelength accuracy test for USP reflection measurement kit

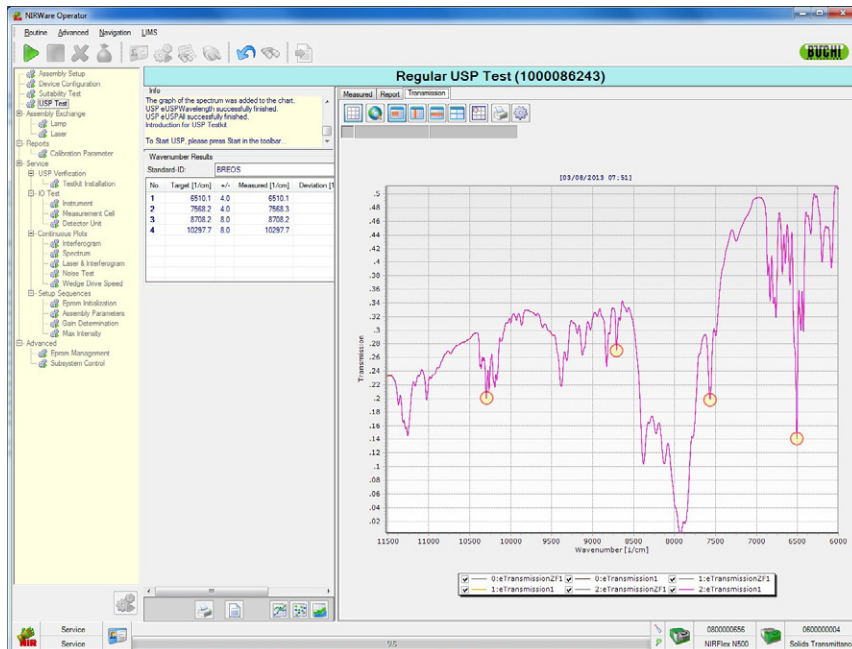


Fig. 6.43: Reflection spectra of the wavelength accuracy test for USP solid transmission measurement kit

For the USP reflection measurement kit, the Reflection tab displays measured reflection spectra in different colors. For the USP solid transmission measurement kit, the transmission tab displays measured transmission spectra in different colors. The legend under the graphic indicates which spectrum is represented by which color. By deactivating or reactivating the corresponding checkboxes you can determine which spectra are displayed. You can now move the cursor over the spectra. In the fields under the iconbar the current X- and Y-values of the cursor position are indicated. Via the iconbar the spectra can be edited, printed and displayed in different views.

6.7 Printing and signing the test data

6.7.1 Printing spectra

ATTENTION

If you want to print the spectra or other displayed plots you have to do this directly after the tests have been carried out. When the NIRWare Operator is closed and opened again afterwards, the spectra are no longer available. They are saved to the database and can be recalled and displayed only with NIRCal.

To print the plots, proceed as follows:

- Have the plots you want to print out shown in the graphic and report area by clicking the corresponding icon in the iconbar.
- Now click the print icon in the iconbar for graphic display.
- File the print-outs of the plots according to your SOP.

6.7.2 Printing the USP Protocol

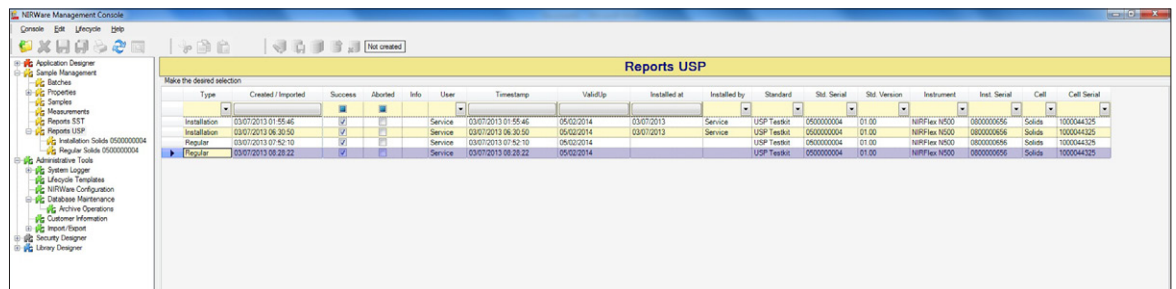
To print the protocol directly from the NIRWare Operator, proceed as follows:

- Select the graphic and report area by clicking the **Report** tab in one of the graphic displays.
- Now click the print icon.
- Sign the USP Protocol and file it according to your SOP.

The USP Protocol is automatically saved in the database and can be accessed via the NIRWare Management Console.

For this purpose, proceed as follows:

- Open the NIRWare Management Console by clicking the corresponding start icon on the desktop.
- Log in with your user name and password and click the green arrow.
- Select **Sample Management > Reports USP**. The following dialog opens:



Type	Created / Imported	Success	Aborted	Info	User	Timestamp	ValidUp	Installed at	Installed by	Standard	Std. Serial	Std. Version	Instrument	Inst. Serial	Cell	Cell Serial
Installation	03/07/2013 01:55:45	✓			Service	03/07/2013 01:55:45	05/02/2014	03/07/2013	Service	USP Testkit	0500000004	01.00	NIRFlex N500	0800000056	Solids	1000044325
Installation	03/07/2013 06:30:50	✓			Service	03/07/2013 06:30:50	05/02/2014	03/07/2013	Service	USP Testkit	0500000004	01.00	NIRFlex N500	0800000056	Solids	1000044325
Regular	03/07/2013 07:52:10	✓			Service	03/07/2013 07:52:10	05/02/2014	03/07/2013	Service	USP Testkit	0500000004	01.00	NIRFlex N500	0800000056	Solids	1000044325
Regular	03/07/2013 08:25:22	✓			Service	03/07/2013 08:25:22	05/02/2014	03/07/2013	Service	USP Testkit	0500000004	01.00	NIRFlex N500	0800000056	Solids	1000044325

Fig. 6.44: Reports USP in NIRWare Management Console

In the table a list with all available USP reports is shown. To open the report you need, double-click it in the list. The following dialog opens:

NIRWare Management Console

Console Edit Lifecycle Help

Application Designer
Sample Management
Batches
Properties
Samples
Measurements
Reports SST
Reports USP
Installation Solids 0500000004
Regular Solids 0500000004
Administrative Tools
System Logger
Lifecycle Templates
NIRWare Configuration
Database Maintenance
Archive Operations
Customer Information
Import/Export
Security Designer
Library Designer

Regular Solids 0500000004

1 /2

SAP CRYSTAL REPORTS®

Main Report

BUCHI

USP Protocol
(in Compliance with PhEur)

USP Test Number : 5
Software : NIRWare USP Module Version
Instrument : NIRFlex N500 Version : 01.02 Serial : 0800000656
Measurement Cell : Solids Version : 01.02 Serial : 1000044925
Cell Option : USP Test Kit
User : Service
Date : 03/07/2013 07:52:10
Standard Set : USP TestKit
Article : 48436
Serial No. : 0500000004 Version : 01.00
Valid to : 05/02/2014

Photometric Linearity / Test at 3 Wavenumbers

Number of scans : 16
Test Wavenumber : 5000 cm⁻¹

Standard -ID	Target [Abs]	Measured [Abs]
GS 80	0.045	0.053
GS 40	0.328	0.321
GS 20	0.583	0.581
GS 10	0.876	0.922
GS 05	1.120	1.117

Slope: 1.000 ± 0.050 act. = 1.013 **passed**
Intercept: 0.000 ± 0.050 act. = 0.001 **passed**
Correlation coef.: 0.999

Test Wavenumber : 6250 cm⁻¹

Standard -ID	Target [Abs]	Measured [Abs]
GS 80	0.055	0.054
GS 40	0.346	0.341
GS 20	0.612	0.601
GS 10	0.903	0.957
GS 05	1.172	1.166

Slope: 1.000 ± 0.050 act. = 1.011 **passed**
Intercept: 0.000 ± 0.050 act. = 0.001 **passed**
Correlation coef.: 0.998

Test Wavenumber : 6330 cm⁻¹

Standard -ID	Target [Abs]	Measured [Abs]
GS 80	0.062	0.072
GS 40	0.361	0.359
GS 20	0.645	0.624
GS 10	0.939	0.998
GS 05	1.238	1.229



Slope: 1.000 ± 0.050 act. = 1.008 **passed**
Intercept: 0.000 ± 0.050 act. = 0.002 **passed**
Correlation coef.: 0.998

Report created at : 03/07/2013 09:06:54 Page 1 of 2

Current Page No.: 1 Total Page No.: 2 Zoom Factor: Whole Page

Service Service

Fig. 6.45: Reports USP in the NIRWare Management Console

The stored reports can be printed out via the default printer using the printer icon  or exported to other formats (e.g. pdf, csv, txt, etc.) by clicking the icon .

7 Maintenance

This chapter gives instructions on all maintenance work to be performed in order to keep the USP Performance Verification Kit in good working condition.



WARNING

Danger of eye irritation.

Some of the powders inside the BREOS Standard are irritating to eyes, respiratory system and skin. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing. Never open the BREOS Standard.



ATTENTION

Never bring the standard windows into contact with hard objects so that the window surface is not scratched. Handle the standards with care.

NOTE:

In case of any damage or resistant contamination on the standard send it back to Büchi for maintenance and revalidation.

7.1 Cleaning

The standard is made of very expensive materials and has to be handled with extreme care. The standard comes in an aluminium housing with a special glass window. The housing can be cleaned using optical tissue with some drops of clean alcohol.

7.2 Recertification of the USP test kit

The Standard Certificate is valid for 2 years. After that time the USP Performance Verification Kit has to be sent back to Büchi Labortechnik AG for recertification. The recertification is performed to confirm that the USP test kit is within the acceptable values and valid compared to the first standard certificate. The target values used for the regular USP test result from the calculated target values of the first certificate.

The recertification of the USP Performance Verification Kit will be delivered back to the customer with:

- a CD containing the xml file of the first standard certification and the xml file of the latest recertification.
- a hardcopy of the standard recertification document, see details in fig 7.1

Standard Recertification					
Büchi Labortechnik AG, CH-9230 Flawil, Switzerland					
Standard Set Serial No: 0400000001 (01.00)					
measured on NIRFlex N500, #0400000013(V00.01) / Solids, #0500000158(V01.01)					
Article:	48426	Grey Standards	[1/cm]	[Abs]	[Abs] SD
1. Certificate Date:	07/06/2010				1. Certificate (measured)
Valid until:	07/01/2012				
Wavenumber Standard:					
BREOS values 1. Certificate [1/cm]	BREOS (measured) [1/cm]	SD			
8'380.3	8'380.0 ± 8	± 0.04			
8'510.2	8'510.2 ± 4	± 0.00			
5'943.7	5'943.5 ± 4	± 0.00			
5'687.9	5'688.1 ± 4	± 0.01			
5'413.0	5'412.9 ± 4	± 0.01			
5'073.2	5'073.1 ± 4	± 0.00			
			GS 80	5000 0.055	0.055 ± 0.0006
				6250 0.066	0.066 ± 0.0006
				8330 0.075	0.074 ± 0.0005
			GS 40	5000 0.321	0.320 ± 0.0010
				6250 0.342	0.341 ± 0.0009
				8330 0.361	0.360 ± 0.0008
			GS 20	5000 0.581	0.581 ± 0.0017
				6250 0.602	0.602 ± 0.0014
				8330 0.626	0.626 ± 0.0012
			GS 10	5000 0.918	0.917 ± 0.0013
				6250 0.956	0.954 ± 0.0010
				8330 0.998	0.996 ± 0.0008
			GS 05	5000 1.109	1.111 ± 0.0012
				6250 1.160	1.162 ± 0.0010
				8330 1.216	1.223 ± 0.0010
Flawil, 07/01/2010					
Quality Assurance Manager				BÜCHI	

Fig. 7.1: Example of a Standard Recertification for the USP reflection measurement

BREOS values 1. Certificate shows the BREOS values of the first certificate

BREOS (measured) shows the BREOS values measured in the production during recertification

SD shows the standard deviation of the BREOS measurement during recertification

Gray Standards 1. Certificate shows the gray standard values from the first certificate

Gray Standards (measured) shows the gray standard values measured in the production during recertification

SD shows the standard deviation of the gray standards' measurement during recertification

There are two ways to handle the recertification data after receiving the recertified USP test kit depending on your corresponding situation. Please follow the procedures that are related to your situation.

When the first standard certificate already exists in the database

- Open the NIRWare Management Console, select **Administrative Tools > Import/Export > Import General**.
- Import the xml file of the recertification from the folder “Recertification” in the delivered CD. Now the USP regular test is ready to be performed. For this purpose, refer to the description in chapter 6.

NOTE

The import of standard recertification data does not require a reinstallation of the USP test kit.

When the first standard certificate does not yet exist in the database

The installation procedure of the USP test kit has to be performed first by the service engineer or a trained person before performing a regular test.

- Import the USP installation file of the first standard certificate by opening the NIRWare Management Console and selecting **Administrative Tools > Import/Export > Import General** and then choosing the corresponding file.
- Now import the latest Standard Recertification xml file by opening the NIRWare Management Console, selecting **Administrative Tools > Import/Export > Import General** and then choosing the corresponding xml file from the folder “Recertification” in the delivered CD. Now the USP regular test is ready to be performed. For this purpose, refer to the description in chapter 6.

NOTE

The import of standard recertification data does not require a reinstallation of the USP test kit.

If you try to import the latest Standard Recertification xml file in the NIRWare Management Console without the first standard certificate available in the database, the following warning message will appear:

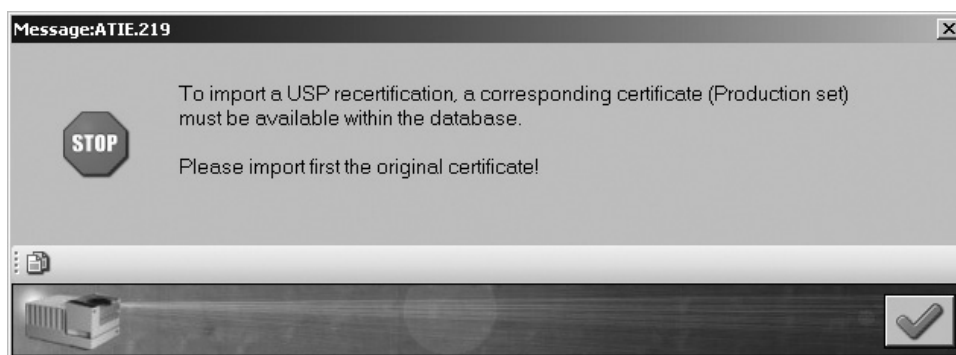


Fig. 7.2: Warning message when importing the USP recertification without the first certificate

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Quality in your hands