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1 About this manual

This manual describes the NIRFlex N-500 including its standard software and provides all information required for its safe operation and to maintain it in good working order.

It is addressed to laboratory personnel and operators in particular.

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

No technical modifications may be made to the instrument without the prior written agreement of Buchi. Unauthorized modifications may affect the system safety or result in accidents. Technical data are subject to change without notice.

NOTE

The symbols pertaining to safety are explained in section 2.

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The English manual is the original language version and serves as basis for all translations into other languages. If you need another language version of this manual, you can download available versions at www.buchi.com.

1.1 Trademarks

The following product names and any registered and unregistered trademarks mentioned in this manual are used for identification purposes only and remain the exclusive property of their respective owners:

- NIRFlex® is a registered trademark of BÜCHI Labortechnik AG
- NIRCal[®] is a registered trademark of BÜCHI Labortechnik AG
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1.2 Abbreviations

EMA: European Medicines Agency *Ph. Eur.*: European Pharmacopoeia *FDA*: Food & Drug Administration *MTBF*: Mean time between failures *NIR*: Near infrared *PMMA*: Polymethylmetacrylat *S/N*: Signal to noise ratio *USP*: United States Pharmacopeia

2 Safety

This section introduces the safety concept of the instrument and contains general rules of behavior and warnings from direct and indirect hazards concerning the use of the product. For the users safety, all safety instructions and safety messages in the individual sections shall be strictly observed and followed. Therefore, the manual must always be available to all persons performing any tasks described herein.

2.1 User qualification

The instrument may only be used by laboratory personnel and other persons who on account of training and professional experience know the potential dangers that can develop when operating the instrument.

Untrained personnel, or persons who are currently being trained, require careful supervision by a qualified person. This Operation Manual serves as a basis for training.

2.2 Proper use

The NIRFlex N-500 has been designed and built as a rugged analysis instrument. It serves to determine the matter and concentration of substances in samples above its measuring tolerance limit (no trace analysis). Thanks to its ruggedness, the instrument is suitable for use on most production floors (at-line, near-line). For suitable environmental conditions see tables of technical data.

The NIRFlex N-500 system can be applied for the following tasks:

Qualitative analysis

- Differentiation of chemically different substances (e.g. raw material testing of incoming goods);
- Differentiation of chemically similar substances or substance grades.

Quantitative analysis

• Determination of quantifiable product properties such as concentrations or physical parameters (viscosity, particle size).

2.3 Improper use

Any other use than the one stated above and any application that does not comply with the technical data is considered to be improper use. Improper use can cause hazardous situations for the operator and/or for the instrument and might cause consequential property damage.

The operator bears the sole risk for any damages or hazards caused by improper use!

In particular, the following uses shall not be permitted

- Installation and use in environments where explosion protection is required
- User repair and maintenance work other than described in this manual
- Reuse of sample material for production that has been in direct contact with non-food safe materials and surfaces

2.4 Safety warnings and safety signs used in this manual

DANGER, WARNING, CAUTION and NOTICE are standardized signal words for identifying levels of hazards and risks related to personal injury and property damage. All signal words, which are related to personal injury are accompanied by the general safety sign.

For your safety it is important to read and fully understand the table below with the different signal words and their definitions!

Sign	Signal word	Definition	Risk level
	DANGER	Indicates a hazardous situation which, if not avoided, will result in death or serious injury.	****
	WARNING	Indicates a hazardous situation which, if not avoided, could result in death or serious injury.	★★★ ☆
	CAUTION	Indicates a hazardous situation which, if not avoided, may result in minor or moderate injury.	****
no	NOTICE	Indicates possible property damage, but no practices related to personal injury.	★☆☆☆ (property damage only)

Supplementary safety information symbols may be placed in a rectangular panel on the left to the signal word and the supplementary text (see example below).

Space for	SIGNAL WORD
supplementary	Supplementary text, describing the kind and level of hazard/risk seriousness.
safety	List of measures to avoid the herein described, hazard or hazardous situation.
information	•
symbols.	•

Table of supplementary safety information symbols

The reference list below incorporates all safety information symbols used in this manual and their meaning.

Symbol	Meaning
	General warning
	Electrical hazard
	LASER emission

Symbol	Meaning
EX	Explosive gases, explosive environment
	Hot item, hot surface
<u><u></u></u>	Device damage
	Fragile components
	Unplug device
	Wear protective goggles
(III)	Wear protective gloves
	Do not dispose as unsorted household waste!

Additional user information

Paragraphs starting with NOTE transport helpful information for working with the device/software or its supplementaries. NOTEs are not related to any kind of hazard or damage (see following example).

NOTE

Useful tips for the easy operation of the instrument/software.

2.5 Product safety

The NIRFlex N-500 has been designed and built in accordance with state-of-the-art technology, at the time of development. Safety warnings in this manual (as described in section 2.4) serve to make the user alert to and avoid hazardous situations emanating from residual dangers by giving appropriate counter measures.

However, risks to users, property and the environment can arise when the instrument is damaged, used carelessly or improperly.

Always follow laboratory safety rules. E.g. wear personal protective equipment such as protective eye googles, protective clothing and gloves when working with the instrument.

2.5.1 General hazards

The following safety messages show hazards of general kind which may occur when handling the instrument. The user shall observe all listed counter measures in order to achieve and maintain the lowest possible level of hazard.

NOTE

The instrument contains a laser for wavenumber calibration. When closed the instrument is considered a Class 1 laser product (DIN Standard: GZS values DIN VDE 0837). When the housing is open, the instrument is considered a Class 3R laser product.

Additional warning messages can be found whenever actions and situations described in this manual are related to situational hazards.





WARNING

A

Serious eye damage by unfiltered LASER emission at open housing.

• Do not operate the instrument with open or damaged housing



Caution

Minor or moderate injuries by dangerous current.

• Do not spill any liquids over the Measurement Cell connector of the instrument

	NOTICE
15	Risk of instrument damage by mechanical shocks.
Do not move the instrument when it is active	
	Do not drop the instrument or its components
	Keep external vibrations away from the instrument
	Do not transport the instrument without transportation lock engaged

2.5.2 Warning labels on housing and assemblies

The following warning sticker(s) can be found on the housing or assemblies of the NIRFlex N-500:

Symbol	Meaning	Location
	Hot item, hot surface	Inside housing, next to lamp modules
	LASER emission	Sticker at the rear side Sticker inside housing, at the LASER module
	Electrical hazard	HV supply LASER

Device labels	Meaning	Location
This device complies with Part 15 of the FCC Rules and the Radio Interference Regulation of the Canadian DOC. Operation is subject to the following two con- ditons: (1) This device may not cause harmful interference and (2) this device must accept any interference received including interference that may cause undesired operation.	FCC declaration	Sticker at the rear side
ACHTUNG! Vor Oeffnen des Gehäuses Netzstecker ziehen! WARNING! Disconnect power supply before removing cover!	See text.	Sticker at the rear side
LASER CLASS 1 LASER KLASSE 1	LASER emission class	Sticker at the rear side
Don't use for powering other devices Use only for connecting NIRFlex Measurement Cells	See text.	Sticker on top of the housing, near the Measure- ment Cell connector.

Device labels	Meaning	Location
LASER RADIATION AVOID EXPOSURE TO BEAM CLASS 3R LASER PRODUCT (IEC) CLASS IN LASER PRODUCT (CDRH)	LASER emission	Sticker inside housing, at the LASER module

2.5.3 Personal protective equipment

Safety precautions and personal protective equipment might be necessary to comply with industrial safety standards in some working environments. However, for standard operation with the instrument, no additional protective equipment is necessary.

2.5.4 Built-in safety elements and measures

The instrument is equipped with the following safety elements:

- One outer filter elements with covering frame
- Multiple magnet sensors to detect opening of any housing element
- Multiple overtemperature sensors to monitor thermal spots inside the instrument
- Transportation lock of interferrometer

2.6 General safety rules

Responsibility of the operator

The head of the laboratory is responsible for training his/her personnel.

The operator shall inform the manufacturer without delay of any safety-related incidents which might occur during operation of the instrument or its accessories. Legal regulations, such as local, state and federal laws applying to the instrument or its accessories must be strictly followed.

Duty of maintenance and care

The operator is responsible for the proper condition of instrument. This includes maintenance, service and repair jobs that are performed on schedule as described in this manual. Any work, not explicitly described in this manual must be performed by authorized and trained personnel (e.g. service technicians) only.

Spare parts to be used

Use only genuine consumables and spare parts for maintenance to assure good system performance, reliability and safety. Any modifications of spare parts or assemblies are only allowed with the prior written permission of the manufacturer.

Modifications

Modifications to the instrument are only permitted after prior consultation 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 section introduces the reader to the instrument specifications and the list of materials used.

3.1 Materials used

Materials used		
Component	Material designation	
Base plate	Aluminium / stainless steel	
Wedges	TeO ₂	
Polarizers	Glass	
Housing	Polyurethane foam	
Detector	InGaAs	
Magnets	NdFeB	
Base plate Wedges Polarizers Housing Detector Magnets	Aluminium / stainless steel TeO ₂ Glass Polyurethane foam InGaAs NdFeB	

3.2 Technical data basic instrument

3.2.1 Basic instrument

Technical data basic instrument	
Dimensions housing (W x H x D)	350 x 250 x 450 mm
Electric power supply	100–230 VAC ± 10 %, 50/60 Hz, 350 W
Ambient conditions	< 80 % relative humidity for T < 31 °C, linear decreasing
	to 67 % at 35 °C, max. 2500 m, for indoor use only
Ambient temperature	$5{-}35~^{\circ}\text{C}$ ($25 \pm 5~^{\circ}\text{C}$ recommended)
Pollution degree	2
Installation category	II
Spectral range	800 - 2500 nm (default 1000 - 2500 nm)
	12'500 - 4'000 cm ⁻¹ (default 10'000 - 4'000 cm ⁻¹)
	(if not specified otherwise for measuring cell)
Resolution	8 cm ⁻¹ (with boxcar apodisation)
Type of interferometer	Polarisation interferometer with ${\rm TeO}_{_2}$ wedges
Wavenumber accuracy	\pm 0.2 cm $^{\text{-1}}$ (measured with HF gascell at an ambient
	temperature of 25 °C \pm 5 °C)
S/N	10000 (peak-to-peak noise of a linear corrected baseline
	between 5600–6000 cm ⁻¹ , measured with NIRFlex
	Liquids, 2 x 64 scans, Blackman apodisation)
Number of scans/sec.	2–4
Analog digital converter	24 bit
Type of lamp/lifetime lamp (MTBF)	Tungsten halogen lamp / 12000 h (2 x 6000 h)
Type of laser	12 VDC HeNe, wavelength at 632.992 nm
Ethernet connection	100 Mbit/s

3.3 Non-fiber optic measuring cells

3.3.1 NIRFlex Solids

Technical data NIRFlex Solids		
Detector	extended range InGaAs (temperature controlled)	
Electric power supply	100 - 230 VAC ± 10%, 50/60 Hz, 20 W	
Ambient conditions	< 80 % relative humidity for T $<$ 31 °C, linear decreasing to 67 % at	
	35 °C, max. 2500 m, for indoor use only	
Ambient temperature	$5-35~^\circ\text{C}$ ($25\pm5~^\circ\text{C}$ recommended)	
Pollution degree	2	
Installation category	l	

3.3.2 NIRFlex Solids Transmittance

Technical data NIRFlex Solids Transmittance	
Detector	InGaAs (temperature controlled)
Electric power supply	100 - 230 VAC \pm 10%, 50/60 Hz, 20 W
Ambient conditions	< 80 % relative humidity for T $<$ 31 °C, linear decreasing
	to 67 % at 35 °C, max. 2500 m, for indoor use only
Ambient temperature	$5-35~^{\circ}\text{C}$ ($25 \pm 5~^{\circ}\text{C}$ recommended)
Pollution degree	2
Installation category	II
Spectral range	12'500 - 6'000 cm ⁻¹
	(recommended range 11'520 - 6000 cm ⁻¹)
	800–1660 nm (recommended range 870 - 1660 nm)
Photometric dynamic range	0 - 6 AU

Technical data NIRFlex Solids Transmittance (cont.)	
Photometric linearity	The "addition of filter technique" has been used. A 2% transmission filter and a wavelength standard (Rare Earth Oxide mixture) were used for the addition. Both filters were measured individually and together in series. The addition of the individual measurements has been compared with the measurement of both filters ihn series. The difference had been $< 2 \times 10^{-7}$ T at 7'876 cm ⁻¹
Typical signal-to-noise ratio	rms for spectral segments of 300 cm ⁻¹ in the range of 11'000 - 6'500 cm ⁻¹
open beam	mean 2 x 10 ⁻⁵ AU (16 scans; Blackman apodization)
5 mm white standard	mean 10 x 10 ⁻⁵ AU (64 scans; Blackman apodization)

3.3.3 NIRFlex Liquids

Technical data NIRFlex Liquids	
Detector	extended range InGaAs (temperature controlled)
Electric power supply provided from NIRFlex N-500	100–230 VAC \pm 10 %, 50/60 Hz, 250 W
Ambient conditions	< 80 % relative humidity for T $<$ 31 °C, linear decreasing to 67 % at 35 °C, max. 2500 m, for indoor use only
Ambient temperature	$535~^\circ\text{C}$ ($25 \pm 5~^\circ\text{C}$ recommended)
Pollution degree	2
Installation category	II
Sample temperature range	Ambient temperature plus 10 °C up to 65 °C
Reproducibility of set sample temperature	± 0.5 °C
Temperature overshoot	< 5 °C
Overheating protection, automatic switch-off	T > 90 °C
Diameter of measurement spot	2 mm
Type of cuvettes to be used	Standard cuvettes 12.5 x 12.5 x 45 mm with path- length of 2 mm, when spacers are used, 1, 5, 10 mm cuvettes can be inserted as well
Time needed to reach a stable control of the set temperature	Ambient temperature to 65 °C: 15 min

3.4 Fiber-optic measuring cells and accessories

3.4.1 NIRFlex Fiber Optic Solids

Technical data NIRFlex Fiber Optic Solids	
Detector	extended range InGaAs (temperature controlled)
Electric power supply	100–230 VAC ± 10 %, 50/60 Hz, 20 W
Ambient conditions	< 80 % relative humidity for T $<$ 31 °C, linear decreasing
	to 67 % at 35 °C, max. 2500 m, for indoor use only
Ambient temperature	$5{-}35~^{\circ}\text{C}$ ($25 \pm 5~^{\circ}\text{C}$ recommended)
Pollution degree	2
Installation category	II
Temperature range at probe tip	0 °C–80 °C
Standard length of fiber optic probe	2 m (available up to 5 m)
Accessories	

lechnical data Transflectance adapter (Fiber Optic Solids)		
Max. operating temperature	120 °C	
Material	Transflectance sleeve material: Steel no. 1.4435	
	Spacer ring material: Steel no. 1.4305	
	Transflectance adapter window: Quartz glass	
	(Infrasil 303) sealed with fluorine rubber 0-rings	
Available pathlengths	0.5 mm, 1 mm, 1.5 mm	
Available pathlengths	0.5 mm, 1 mm, 1.5 mm	

3.4.2 NIRFlex Fiber Optic Liquids

Technical data NIRFlex Fiber Optic Liquids	
Detector	extended range InGaAs (temperature controlled)
Electric power supply	100-230 VAC ± 10 %, 50/60 Hz, 20 W
Ambient conditions	< 80 % relative humidity for T < 31 °C, linear decreasing
	to 67 % at 35 °C, max. 2500 m, for indoor use only
Ambient temperature	$535~^\circ\text{C}$ ($25 \pm 5~^\circ\text{C}$ recommended)
Pollution degree	2
Installation category	11
Temperature range of the probe head at the probe tip	0 °C–150 °C
Max. pressure at the probe tip	6 bar
Standard length of fiber optic probe	2 m (available up to 7 m)
Pathlength	2 mm

3.4.3 NIRFlex Fiber Optic SMA

Technical data NIRFlex Fiber Optic SMA	
Detector	extended range InGaAs (temperature controlled)
Electric power supply	100–230 VAC \pm 10 %, 50/60 Hz, 20 W
Ambient conditions	< 80 % relative humidity for T < 31 °C, linear decreasing
	to 67 % at 35 °C, max. 2500 m, for indoor use only
Ambient temperature	$535~^\circ\text{C}$ ($25 \pm 5~^\circ\text{C}$ recommended)
Pollution degree	2
Installation category	11

NOTE

Limit ranges of environmental conditions at the point of measurement highly depend on individual accessories (i.e. process probe, flow cell, etc.).

4 Description of function

This section explains the basic principle of the instrument, shows how it is structured and gives a functional description of the assemblies.

4.1 Functional principle

The NIRFlex N-500 is a modular optical instrument (basic instrument and measuring cell) to determine the matter and concentration of substances in samples.

In detail, the NIRFlex N-500 is a **F**ourier **T**ransformation **N**ear Infrared spectrometer (FT-NIR). It generates an invisible near infrared interferogram beam which interacts with the molecules of a sample, generating a characteristic feedback. The feedback is picked up via a measurement cell by a detector and mathematically processed via Fourier transformation into a spectrum. This spectrum is used to extract the requested material information.

Inside the spectrometer, a laser beam is used as a high-precision wavelength reference to allow best possible reproducibility and accuracy of detection.

Advantages of FT-NIR polarization inteferometer

- Simultaneous measurement of all wavenumbers giving an improved signal-to-noise ratio
- Higher intensity giving an improved signal-to-noise ratio and short measuring times
- Laser as wavenumber reference giving high wavenumber stability and good data transferability
- Single-beam interferometer without typical double-beam divergence for mechanically and temperature stable beam alignment
- More robust design than standard Michelson interferometer

How the interferogram is generated

An interferogram is an interference pattern of phase-shifted beams. The NIRFlex N-500 is a singlebeam polarisation interferometer, generating its interferogram in four steps:

Step 1 - Polarization of the light source output

The polarizer **2** generates a well-defined polarization output of the undefined polarized light, emitted by the light source **1**. Thus, only diagonally polarized light is transmitted.

Step 2 — Beam splitting and orthogonal polarization

The polarized light enters a double refracting block (comparator) **3**. Here, the light is broken down into two, orthogonally polarized components with a small, static phase shift.

Step 3 - Generating the ongoing phase shift

An assembly of two double refracting wedges is arranged after the comparator. Wedge 4 is stationary, while wedge 5 is constantly shifted back and forwards by a fast linear-drive. The movement and the geometric arrangement provides a change of thickness in the light path. This leads to an ongoing phase shift between the light beams.

Step 4 — Beam recombination and interferogram output

A second polarizer 6 converts the phase shifted beams into a single light output with intensity variation – the interferogram.

Reference laser control

The laser deliveres a constant, wavelength-stable beam of 633 nm. The laser beam (1) is coupled into the NIR beam (2) to pass the interferometer before being decoupled (4) and analysed by the "Laser feedback sensor" (5). A fraction of the beam is splitted at the polarizer (2) and picked up by an intensity sensor (3) to monitor the laser beam quality. The position and movement-frequency of the wedge by the linear drive (6) causes an alternating relative phase shift of the laser light. Being reflected by the second polarizer (4), the phase shift generates an alternating amplitude. This alternating amplitude is detected via the the "Laser sensor" (5), giving precise information about velocity and (relative) position of the wedge. The feedback signal is used to control the velocity of the linear drive (6) as well as to set the sampling-points of the NIR interferogram.



Effective NIR light path

0	NIR light source assembly with spare lamp and motorized parabolic reflector
2	First polarizer
3	Comparator (double refracting block)
4	Stationary double refracting wedge
6	Moving double refracting wedge
6	Second polarizer
7	Sample
8	Interferogram detector
Refere	nce laser signal path
1	Laser output window
2	Second polarizator (works as a beam splitter for the laser)
3	Laser output-power sensor
4	First polarizer
5	Laser feedback detector
6	Linear motor for wedge movement

Data processing and interferogram analysis

The NIR light interacts with the sample 🕜 material in different ways, leaving a characteristical fingerprint on the interferogram. At liquids the light is mostly transmitted and at solids reflected. The remaining light is collected by the detector (3). The built-in computer further processes the raw signal.

Process steps	Result
Signal preprocessing	Interferogram
Fourier transformation	Raw spectrum
Signal background correction	Spectrum of sample
Chemometric analysis of the spectral data	Sample analysis
Display of the result via NIRWare Operator on the attached monitor	Sample analysis is displayed

4.2 Measuring cells and their modes

The NIRFlex N-500 system is of modular design. The interferometer is located in the basic instrument. Different measuring cell modules for different kinds of sample materials can be easily attached to the basic intrument. To choose the best measuring cell for a specific range of samples the optical properties of the sample material must be know.

Measuring cell mat	rix		
Sample characteristics	Section	Measuring cell	Typical application
(for NIR light)			
Diffuse reflection	4.2.1	NIRFlex Solids;	Predominantly non-translucent
		NIRFlex Fiber Optic Solids;	materials such as most tablets,
		NIRFlex Fiber Optic SMA*	cereals and powders
Transmission	4.2.3	NIRFlex Liquids;	Translucent and transparent liquids
		NIRFlex Fiber Optic Liquids	
		NIRFlex Fiber Optic SMA*	
Diffuse transmission	4.2.4	NIRFlex Solids Transmittance	Predominantly translucent solids
			such as some tables, crystal
			powders and other light conducting
			materials
Transflectance	4.2.5	NIRFlex Fiber Optic Solids/NIRFlex	Solids with weak diffuse reflection
		Solids with Transflectance Adapter;	and low transmission rate charac-
		NIRFlex Fiber Optic SMA*	teristics
		*Depending on the custom accessory	

and measurement setup

4.2.1 Transflectance mode

Translucent and opaque liquids can be analyzed via transflectance mode. The light penetrates the liquid, is diffusely reflected by the reference plate and passes the sample a second time. The transflected rays contain the spectral information of the sample.

Factors that can influence the measurement:

- Not enough sample material available
- Air bubbles in the measurement path and under the transflectance cover
- Sample not homogeneous or representative
- Temperature of sample material
- Sample cup or transflectance cover inadequate (e.g. cup material and thickness, blind spots, dirt etc.)





NIRFlex Fiber Optic Solids/SMA:



NOTE

- With special accessories, reflection-mode probes can be equipped for transflectance.
- The samples should either be measured at a constant temperature or the temperature influences must be considered during calibration.
- Recommended number of scans: Qualitative calibration: 4–16; quantitative calibration: 16–32
- Most liquid sample materials can be constantly analyzed with the help of a flow-cell adapter.

4.2.2 Diffuse reflection mode

Most non-translucent materials (e.g. solids such as powders, pellets and cereals) can be analyzed via diffuse reflection. The NIR light penetration is limited by the sample material. It interacts with the sample, is refracted and diffusely reflected into the sensor. The reflected rays contain the spectral information of the sample.



Factors that can influence the measurement:

- Not enough sample material available
- Sample not homogenious or representative
- Humidity of sample material
- Temperature of sample material
- Sample cup inadeqate (e.g. cup material and thickness, blind spots, dirt etc.)

NOTE

- The samples should either be measured at a constant temperature or the temperature influences must be considered during calibration.
- Coarse grained samples should be milled before carrying out the measurement. Recommended number of scans: Qualitative calibration: 4–16; quantitative calibration: 16–32; granules: 64.

4.2.3 Transmission mode

NIR light is sent through a defined pathlength of sample material (e.g. in a cuvette). The transmitted light contains the spectral information. This is the preferred method for testing liquids.



4.2.4 Diffuse transmission mode

The diffuse transmission mode is a mixture of "diffuse reflection" and "transmission" mode.

The NIR light penetrates the sample:

- is refracted
- diffusely reflected
- diffusely transmitted

The transmitted rays contain the spectral information of the sample.



4.3 Basic instrument

The basic instrument consists of the spectrometer unit with the interferometer, the lamp unit with two lamp modules, the laser unit and electronic boards. The Instrument Software (embedded software) controls the spectrometer and communicates with the PC running NIRWare and with the measuring cell. It controls all actors (stepper motor, heating, etc.) and sensors (light barrier, temperature sensor, etc.) in the measuring cell. The sensor is part of the measuring cell.



- Main switch
- ② Primary fuses
- ③ Power supply socket
- (4) Type plate with serial number

- (5) Ethernet connection
 (a) Interfaces for service interview
- (a) Interfaces for service intervals (USB 1, USB 2, KBD, MS, VGA, COM)
 (7) Ventilation filter

4.3.1 Lamp unit

The lamp unit comprises two lamp modules:

- A primary lamp module, which is normally in use.
- A secondary lamp module, which is only in use during failure of the primary lamp module.

When the NIRWare software detects a lamp failure, it automatically switches to the secondary lamp module. Until the primary lamp module has been exchanged the spectrometer measures spectra with the secondary lamp module.

The operator will be reminded to exchange the primary lamp module, which he can easily do by himself, see section 7.4. The secondary lamp module can only be exchanged by service personnel. The hours of operation are logged separately for each lamp module. The lifetime of the lamps is about 6000 h each.

4.3.2 Laser unit

The laser unit comprises:

- Laser tube
- High voltage power supply

The intensity of the laser light is continuously checked. When it falls below a certain threshold, the operator has to change the laser, see section 7.5. The hours of operation are logged. The typical lifetime of a laser is about 20000 h.

4.3.3 Standard wheel



Standard wheel

Every NIRFlex N-500 is equipped with a standard wheel with standards that are used for the System Suitability Test (SST).

The standards within the wheel have the following functions:

- Wavelength standard (PMMA) to check the wavelength accuracy.
- Open beam for normal measurements.
- Five different grey standards to check the linearity.

The software is designed for the automatic use of the standard wheel, i.e. the operator does not have to start one of the above mentioned tests manually, to ensure proper measurement of the SST and NADIA.

4.4 NIRWare software suite

NIRWare is the interface program suite between the instrument and the operator. All program parts are hosted on an external PC. The PC communicates with the spectrometer via internal network adapter.

NOTE

- A second network interface at the PC is recommended to connect the NIRFlex N-500 to a local PC network, e.g. for data backup or to communicate with enterprise resource planning etc.
- It is recommended to turn off all power saving options.

About the external PC

The external PC must run Windows® as operating system and meet the system requirements (see NIRWare manual). The NIRWare software suite must be installed successfully on the computer. The database might be installed on a network drive, to be accessible by different spectrometer.

4.4.1 Available NIRWare packages

Two predefined NIRWare packages are available for the NIRFlex N-500:

Available NIRWare packages					
	Basic	Advanced			
NIRWare Operator	Х	х			
NIRWare Management Console					
Application Designer	Х	х			
Sample Manager	Х	Х			
Administrative Tools	Х	х			
Security Designer	Х	х			
Library Designer		х			
Regulatory Kit		х			

Each package includes the following program elements by default:

- Operator interface, to run routine analysis
- Administrative tools
- Report templates
- Comprehensive database with analysis data and other

The NIRWare Basic package offers the minimum requirements to use the NIRFlex N-500 FT-NIR system. This package is recommended for routine analyses with pre-calibrated applications or as satellite systems in a large application network.

The NIRWare Advanced package offers all components to comply with pharmaceutical requirements based on a lifecycle concept. It is recommended to complete this package with the NIRCal Chemometric Software, which enables the development of own calibrations. Together with the Library Designer a bunch of possibilities for identity control is available.

4.5 About the software

The NIRWare software meets industrial standards in terms of inner structure combined with a compre-hensive and intuitive, wizard guided user interface. It is also ideally suited for many applications in the pharmaceutical, food and feed industry.

Advanced software and system management is done with the NIRWare Management Console tool.

The NIRWare Management Console combines various software modules:

- NIRWare Application Designer to define NIRWare Operator applications
- NIRWare Sample Manager to administrate all samples and reference values
- NIRWare Administrative Tools for applications and calibrations interchange and other administrative tasks
- NIRWare Security Designer to define users and user groups according to custom security policies

The BUCHI Database Manager is used for any database operations.

Optional:

- NIRWare LIMS to import and export sample information and measurement data
- NIRWare Library Designer is a powerful software module for substance identity control using full spectral comparison. It is designed for the development of spectral libraries tailored to individual user requirements.
- NIRWare Regulatory Kit provides all components to comply with pharmaceutical regulations.
- **Instrument Standardization kit** is used on instruments in a network to ensure that the results among instruments are aligned to each other.

4.5.1 NIRWare Control System Service

The NIRWare Control System Service controls the complete system. It runs in the background as a service. Normally there is no user interaction necessary.

4.5.2 NIRWare software suite

The PC is running the NIRWare Software Suite. NIRWare is the interface between the instrument and the operator. All software tools are included, like Administrative Tools, report templates, elements to create new applications and to run routine analyses, and the database to save all results and data. The outstanding features of NIRWare are its logical structure, an industry-standard design and at the same time an easy to understand user interface.

The NIRWare Software Suite comprises the following modules:

- NIRWare Management Console combines various software modules:
 - NIRWare Application Designer defines the applications performed using NIRWare Operator
 - NIRWare Sampe Manager administers all samples and reference values
 - NIRWare Administrative Tools imports and exports applications and calibrations and is used for other administrative work
 - NIRWare Security Designer is used for the administration of users and user groups and for the definition of security policies
- Optional modules for the NIRWare Management Console:
 - NIRWare Library Designer is a powerful software module for identity control using full spectral comparison. It is designed for the development of spectral libraries tailored to individual user requirements.
 - NIRWare Regulatory Kit provides all components to comply with pharmaceutical regulations.

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4.5.3 NIRCal chemometric software

NIRCal is a modern chemometric software package for use with Buchi FT-NIR Spectrometers. It can be used for qualitative and quantitative applications. For method development a lot of comprehensive pretreatments are available. However, the software is easy to use even for novices. The Calibration Wizard guarantees the development of reproducible calibrations. Complex calibrations and interpretations are greatly simplified by the use of wizards, which automate standard procedures and help to develop calibrations.

4.6 Measuring cells with their add-ons and accessories

The modulated light coming from the interferometer interacts with the sample. The light is then detected in the measuring cell and the data is sent to the basic instrument. The measuring cell has its own power supply and is galvanically isolated from the basic instrument. It can be swapped without switching off the basic instrument and is automatically identified (plug & play).

4.6.1 Operating panel

Each measuring cell is equipped with an operating panel:



Operating panel of a measuring cell

③ STOP button

The START button is used to start a measurement without using a PC keyboard. The STOP button is used to stop a measurement without using a PC keyboard.

An LED informs the user about the current instrument state: **Red**: Instrument is in error state **Green**: Instrument is ready to use **Green flashing**: Instrument is busy measuring **Yellow**: Power is on but the instrument is not yet initialized by the NIRWare software

For a description of the LEDs at the handle of the fiber optic, see sections 4.6.15 and 4.6.17.

4.6.2 NIRFlex Solids

List of NIRFlex Solids add-ons							
	Spinner/Petri dish	Vial add-on	Tablet add-on	XL add-on	Flow cell		
	add-on						
Max. number of samples per sequence	1	6	10	1	1		
Measurement based on diffuse reflection	Х	Х	х	Х	-		
Measurement based on transflectance	Х	-	-	-	х		
Measurement with petri dishes	Х	-	-	-	-		
Measurement with vials	-	Х	-	-	-		
Measurement of tablets	-	-	Х	-	-		
Measurement using small plastic bags	-	-	-	Х	-		
Internal reference	Х	-	-	Х	Х		
External Reference	Х	х	х	Х	Х		
Standardization Kit: for Petri dish and XL add-ons	Х			Х			
Sample dimensions	Spinner 34 mm Petri dish 100 mm	Glass vials 10–15 mm	Tablets 5–10 mm		>0.3 ml		



The NIRFlex Solids is the ideal configuration to measure solid samples like powders, pastes and pellets in diffuse reflection mode. The measuring cell for solids allows the use of different add-ons for specific sample containers.

The software automatically detects the type of add-on in use as soon as the corresponding application is started in the NIRWare Operator.

For a description of the add-ons to be used with the NIRFlex Solids, see section 4.6.3 to 4.6.8.



- (8) Sample
- (9) Petri dish

Functional principle of the NIRFlex Solids

4.6.3 Petri dish add-on for NIRFlex Solids



NIRFlex Solids with petridish add-on

The NIRFlex Solids with the petri dish add-on is the ideal configuration to measure solid samples instandard glass petri dishes. The petri dish add-on also enables to use a transflectance cover to analyze liquids (e.g. solvents, milk, etc.). The transflectance technique is a good compromise for customers, who only analyze liquids occasionally.

To carry out the reference measurement, the user has to place the External Reference (see also section 5.10) onto the sample window. The sample applies for the Standardization kit.

4.6.4 Vial add-on for NIRFlex Solids



NIRFlex Solids with vial add-on

The NIRFlex Solids with the vial add-on is the ideal configuration to measure solid samples in glass vials. This special add-on is equipped with 6 positions for sample vials with a diameter of 10–15 mm. Buchi provides one type of glass vials as an optional accessory for this specific add-on. This built-in External Reference is measured automatically without any interaction of the operator. A measurement sequence consists of a measurement of the reference and afterwards the measurement of the sample positions (1–6), which have been pre-defined in the NIRWare Operator. The actually measured spectrum is set against the last measured reference.

4.6.5 Tablet add-on for NIRFlex Solids



NIRFlex Solids with tablet add-on

The NIRFlex Solids with the tablet add-on is the ideal configuration to measure tablets in diffuse reflection mode. This special add-on is equipped with 10 sample positions for round tablets with a diameter between 5–10 mm.

This built-in External Reference is measured automatically without any interaction of the operator. A measurement sequence consists of a measurement of the reference and afterwards the measurement of the sample positions (1-10), which have been pre-defined in the NIRWare Operator.

4.6.6 XL add-on for NIRFlex Solids



NIRFlex Solids with XL add-on

The NIRFlex Solids with the XL add-on is the ideal configuration to measure solid sample in undefined sample containers, like bags, beakerglasses, etc.

The sample container is placed on the flat add-on directly over sampling window. The sample holder will not rotate during the measurement.

To carryout the reference measurement, the userhas to place the External Reference (see also section 5.10) onto the sample window. The sample applies for the Standardization kit.

4.6.7 XL add-on with iris aperture for NIRFlex Solids

The NIRFlex Solids with the XL add-on with iris aperture is the ideal configuration to measure solid samples in bigger glass vials with an diameter of up to 39 mm. The sample holder will not rotate during the measurement. To carry out the reference measurement, the user has to place the External Reference (see also section 5.10) onto the sample window. The sample applies for the Standardization kit.



XL add-on with iris aperture

4.6.8 XL add-on for B+L sample cups for NIRFlex Solids

The NIRFlex Solids with the XL add-on for B+L (Bran+Luebbe) sample cups is the ideal configuration to measure solid sample in the B+L closed cup. The sample holder will not rotate during the measurement.

NOTE

For reference measurement remove the B+L cup first. Place the centering ring and the External Reference onto the XL add-on and perform the measurement.



XL add-on for B+L sample cups

4.6.9 External Reference or a Standardization kit for a for XL and petri dish add-on





External Reference

Standardization kit

An External Reference or a Standardization kit can be utilized when the XL add-on or the petri dish add-on are used on the NIRFlex Solids.

The reference holder is designed with an opening to insert the External Reference or the Standardization kit into the correct position.

NOTE

The reference holder is needed with the petri dish add-on only.

4.6.10 Spinner add-on

NIRFlex Solids in combination with the Spinner add-on is the ideal configuration to measure smaller solid samples whereas the volume of a petri dish is too big. This add-on can also be used with a transflectance cover to analyze liquids (e.g. solvents or milk). The transflectance technique is suitable for customers, who analyze liquids occasionally.



Spinner add-on

Cuvettes (i.e. Hellma cuvette) or glass vials with a diameter of 34 mm can be used as sample vessel in the motor-driven Spinner. For reference measurement the Spinner can hold the External Reference.

4.6.11 Flow-cell adapter add-on

The NIRFlex Solids with flow-cell adapter add-on is the ideal configuration to measure liquids (e.g. solvents and milk.) in a quartz flow-through cuvette with 1 mm pathlength.



Flow-cell adapter add-on

To connect the flow-cell, silicone tubes with 4 mm inner and up to 7 mm outer diameter can be used. External referencing requires a clean and dry flow-cell. In some applications, referencing can also be done with measurement solvent in the flow-cell.

4.6.12 NIRFlex Solids Transmittance



NIRFlex Solids Transmittance

It is a major request described in the pharmacopoeias to ensure consistency of dosage units; each unit in a batch should have active ingredient content within a narrow range around the label claim. Traditionally these analyses are made using HPLC determining only the active content, which is time consuming and very costly. The speed, efficiency, and cost situation can be very much improved by NIR spectroscopy.



Transmission measurements offer the advantage of collecting the information from the entire cross section of the samples. This is ideal, especially for coated or multilayer tablets. NIRFlex N-500 is optimized for transmission measurements of solid dosage forms, like tablets or capsules.



Detector
 Sensor position and beam path

Sample position

4.6.13 Sample plates for the NIRFlex Solids Transmittance



The autosampler accepts up to thirty samples with a diameter of 4 to 12 mm maximum.

The picture shows an example of a 30-place sample plate.



For samples larger than 12 mm up to 30 mm, 10-place sample plates are available.

The picture shows an example of a 10-place sample plate.



The picture shows the 10-place sample plate with iris aperture for method development.


The picture shows the SST sample plate.

The picture shows an examle of a 30-place sample plate for hard gelatin capsules.

Sample plates of the NIRFlex Solids Transmittance

4.6.14 NIRFlex Liquids



NIRFlex Liquids

The NIRFlex Liquids is the ideal configuration to measure liquids like solvents, oils, etc. in transmission mode using glass cuvettes. The NIRFlex Liquids enables quantitative and qualitative analyses with thermostated conditions of the samples (from room temperature plus 10 °C up to 65 °C.

The NIRFlex Liquids can accommodate up to six cuvettes with pathlengths up to 10 mm. Current and set temperatures are displayed in the user interface.



4.6.15 NIRFlex Fiber Optic Solids



NIRFlex Fiber Optic Solids

The NIRFlex Fiber Optic Solids is the ideal configuration to measure solid samples in their original container. The reflection probe operates with a fiber bundle.

The reflection probe can accommodate a transflectance adapter to analyze liquids as well (e.g. solvents, milk, etc.).

The transflectance technique is a good compromise for customers, who only analyze liquids occasionally.

The handle is equipped with a remote control for initiating the measurements remotely and displaying the results using LEDs on the handle.

The LEDs at the handle of the fiber optic probe indicate the status of the system and the results of the prediction in routine operation.

Applications without calibration (just measuring spectra, e.g. acquisition of calibration spectra)		
-	green LED flashing	measuring
-	green LED on measureme	
red LED on	green LED on	message on monitor

Application with calibration (routine use for predictions) green LED flashing measuring red LED on green LED on message on monitor

 green LED on
 result conforms

 red LED on
 result does not conform

Because the fiber optic probe is used at a certain distance from the spectrometer, the length of the fiber optic cables between the measuring cell (spectrometer) and the probe tip is about 2 m (3 and 5 m versions are available as well).



Functional principle of the NIRFlex Fiber Optic Solids

4.6.16 Transflectance adapter for NIRFlex Fiber Optic Solids

The transflectance adapter is an optional accessory for the NIRFlex Fiber Optic Solids and enables to measure liquid samples, pastes and transparent films.

The transflectance measurement principle is a combination of transmission and diffuse reflection: The light coming from the optical fibers penetrates the liquid sample in the gap between the probe window and the transflectance adapter window (½ pathlength), is diffusely reflected at the white standard layer behind the adapter window, passes the sample liquid in the gap a second time and goes back into the optical fibers.

The transflectance adapter is suitable for the analysis of viscous, non-agressive liquids. Thanks to the reproducible, adjustable pathlength it is suitable both, for the meausurement of slightly and of strongly absorbing samples.

The transflectance technique is a good compromise for customers, who only analyze liquids occasionally.

As the measurements are carried out in the original sample containers, the probe head needs to be dipped into the sample.



Transflectance adapter

4.6.17 NIRFlex Fiber Optic Liquids



NIRFlex Fiber Optic Liquids

The NIRFlex Fiber Optic Liquids is the ideal configuration to measure liquid samples in their original container.

The transmission probe operates with monofibers.

As the measurements are carried out in the original sample containers, the probe head needs to be dipped into the samples.

The handle is equipped with a remote control for initiating the measurements remotely and displaying the results using LEDs on the handle.

The LEDs at the handle of the fiber optic probe indicate the status of the system and the results of the prediction in routine operation.

Applications without calibration	(just measuring spectra,	e.g. acquisition of calibration spectra)	

-	green LED flashing measuring	
-	green LED on	measurement finished
red LED on	green LED on	message on monitor

 Application with calibration (routine use for predictions)

 green LED flashing
 measuring

 red LED on
 green LED on
 message on monitor

 green LED on
 result conforms

 red LED on
 result does not conform

Because the fiber optic probe is used at a certain distance from the spectrometer, the length of the fiber optic cables between the measuring cell (spectrometer) and the probe tip is about 2 m (special versions with longer fiber optic cables are available on request).



4.6.18 NIRFlex Fiber Optic SMA

The NIRFlex Fiber Optic SMA is the ideal configuration to measure both, liquids and solids with external accessories. The SMA-cell can be used to connect mono or multi fibers via its two SMA connectors. Laboratory and immersion process probes in transmission or diffuse reflection mode as well as flow-through cells can be attached.



NIRFlex Fiber Optic SMA

NOTE

- Glas fibers have to be handled with care. Do not bend, pull or twist the fibers! Follow the supplementary instructions of the fiber and accessories. See also section 5.8!
- Depending on the application, distances of up to 100 m can be realized.
- External referencing has to be performed with clean and dry accessory!

5 Putting into operation

This section describes how the instrument is installed and gives instructions on initial startup.

To avoid damage by condensate moisture, special attention is needed when the instrument was delivered at cold temperatures. Do not remove the instrument from its plastic bag for at least two hours to let the instrument acclimatize to the ambient temperature!



NOTE

Inspect the instrument package for damage immediately after delivery. If necessary, prepare a status report immediately and inform customer and your local Buchi representative. Keep the original packaging for future transportation.

5.1 Transportation lock

The NIRFlex N-500 is equipped with a mechanical transportation lock to avoid damage of the interferometer during transportation and shipping by shocks and vibrations. The transportation lock is in "Locked" position at initial delivery.

After system installation and prior use, the interferometer has to be unlocked! See following section for information about unlocking.





Operating the transportation lock

5.2 Requirements concerning the installation site

Basic installation conditions

	DANGER
	Death or serious injuries by use in explosive environments.
	Do not store or operate the instrument in explosive environments
	Remove all sources of flammable vapors
EX	Do not store chemicals in the vicinity of the device

The instrument must be installed:

- indoor only with a clearance of 15 cm minimum to walls and any other objects
- without being exposed to heating or cooling source (e.g. direct sunlight or air conditioning)
- with the transportation lock in unlocked position (see previous section)
- on a stable and horizontal position

NOTE

Because of its weight it is recommended to carry the instrument with two persons.

If the instrument is installed on a laboratory cart, the cart should have large, soft wheels (approx. 10 cm diameter) for better shock absorption at transport on even floors.

To avoid damage by condensate moisture, special attention is needed when the instrument is moved between environments with bigger temperature difference! When moving the instrument from a colder to a warmer environment, let it acclimatize to the ambient temperature before switching it on!



NOTICE

Risk of instrument damage by moisture condensate.

• Let the instrument sufficiently acclimatize at relocation

The electric plug on the measuring cell is only designed to plug-in the measuring cells. Do not connect any other item.

The spectrometer must be placed onto a clean, level and stable support. It is recommended to set up the spectrometer so that it is easily accessible from all sides.

The spectrometer is normally operated from the front. Make sure that ergonomic and back-friendly operation is insured.

5.3 Electrical connection requirements

After the pre-installation procedure (all internal connections have been established and transportation lock is in unlocked position) has been completed successfully, the power plug of the NIRFlex N-500 can be connected to mains.

The used mains circuit has to:

- provide the voltage that is given on the type plate of the instrument.
- be able to handle the load of the connected instruments.
- be equipped with adequate fuses and electrical safety measures, in particular proper grounding.

See also technical data of all components regarding the different minimum system requirements!

	NOTICE
! }	Risk of instrument damage by wrong mains supply.
	External mains supply must meet the voltage given on the type plate
	Check for proper grounding
17	Exchange defective cabling instantly

NOTE

- To cut the power in case of an emergency by unplugging, the instrument or any other item must not block the mains plug! The plug must be able to be pulled out instantly at all time.
- Additional electrical safety measures such as residual current breakers may be necessary to meet local laws and regulations!
- External power switches (e.g. emergency stop switches) must meet IEC 60947-1 and IEC 60947-3 requirements. Such devices must be clearly labeled and accessible at any time.
- External connections and extension lines must be provided with a grounded conductor lead (3-pole couplings, cord or plug equipment). All used power cords must meet the input power requirements.

5.4 Installing the instrument

To install the instrument, proceed as follows:

- Connect the NIRFlex N-500 to the PC using the supplied Ethernet cable.
- Connect the mains cable to the socket.
- Switch on the NIRFlex N-500 via the main switch on the rear of the instrument.
- Start the PC, and install the NIRWare software.
- Set up the communication between NIRFlex N-500 and the PC.

5.4.1 Setting up the Ethernet communication

The NIRFlex N-500 is equipped with one Ethernet (LAN) socket at the rear side.





LAN settings of the NIRFlex N-500

The primary network connector (LAN1) is predefined for direct communication to a PC.

Standard configuration of the Spectrometer:

- Static IP-address 192.168.1.1
- Subnet mask 255.255.255.0

NOTE

- The default IP-address of the spectrometer can be changed to a customer specific static IP-address provided by the local IT. This configuration is done within the Service Tools and described in the NIRWare Online Help and the corresponding PDF-file.
- The spectrometer's IP-address can be reset to default (192.168.1.1) by pushing and holding both, START and STOP button, at the measuring cell during the power-up procedure of the NIRFlex N-500 for about 50 seconds.

LAN setup of the PC

To establish communication, set up the IP of the connected PC in accordance to the local network environment or via direct link as listed below.

IP-configuration of the connected PC (direct link):

- Static IP-address 192.168.1.2
- Subnet mask 255.255.255.0

5.4.2 Network integration

There are various possibilities how the NIRFlex N-500 can be installed and used. In the following, two examples are given:



All data on one PC via direct link

The PC with the NIRWare database is directly connected to the NIRFlex N-500. All data remain on that specific PC.



NIRWare database on central company server via indirect link

The NIRWare database runs on a central company server. The Administrator has access to the database on that server. The data measured by the Operator are collected on the database of the central company server.

NOTE

Other setups i.e. with the NIRFlex N-500 directly attached to a local network can also be realized.

5.5 Installing the measuring cells

All different measuring cells are provided with a plug connection.



Installing a measuring cell

To install a measuring cell, proceed as follows:

- Place the measuring cell vertically into the provided space.
- Carefully applying pressure to the measuring cell, make sure that the plug connection catches.
- Make sure it is seated well by means of the centerning pins at the NIRFlex N-500.
- Subsequently, fasten the measuring cell with the fixating screw at the bottom of the NIRFlex N-500.

NOTE

When using the NIRFlex Solids Transmittance cell, put the sample plate onto the cell by inserting the central hole into the shaft. The guiding pin must be inserted into the corresponding hole (next to the handle).





5.6 Installing the cover of the NIRFlex Solids Transmittance

Insert the tilted cover ① and hook its left hinge onto to the bushing pin (hinge counterpart) at the rear left hand side of the measuring cell. Tilt the cover back into a straight position ②.



Keep the right hinge of the cover in a matching position with the bushing screw. Fasten the screw (3) with a few turns (the screwdriver is part of the scope of delivery). Open and close the cover several times to verify the correct installation.



Additional handling instructions



- Install the measuring cell with the top cover closed to avoid mechanical stress at the hinges!
- Keep the top cover closed during a measurement to keep out scattered ambient light.
- Open and close the top cover carefully.



Additional handling instructions

At closing: A small bumper avoids hard impact to the cover.

At opening:

Do not release the top cover until it is in fully open position! The hinges might break-away.

5.7 **Preparing the transflectance adapter**

5.7.1 Adjusting the pathlength of the transflectance adapter to the probe head

When you receive the transflectance adapter with a new instrument:

When you receive your transflectance adpater together with a new NIRFlex N-500 instrument, the pathlength has already been adjusted to the probe head by the manufacturer.

When you receive the transflectance adapter as additional accessory for an instrument already installed:

As the probe head dimensions may vary slightly you need to adjust the transflectance adapter to the probe head when you receive it as additional accessory for an instrument already installed. A correct adjustment is necessary for the portability of spectra.

To adjust the pathlength of the transflectance adapter to the probe head, the spacer ring 1 mm is used. The other spacer rings have been manufactured to fit accordingly.

Normally, the transflectance adapter is delivered with the adjustment ring and the spacer ring 1 mm mounted. If this is the case, proceed with section 5.7.2.



If this is not the case, proceed as follows:

• Slide the adjustment ring over the transflectance sleeve and turn it on the thread as far as possible.

- Now slide the 1 mm spacer ring over the transflectance sleeve, so that the pin on the inner surface of the spacer ring fits into the groove in the adjustment ring.
- Place the 1 mm spacer disk in the center of the transflectance adapter window.



- Slide the transflectance adapter over the probe head and orient it so that the pin at the probe head is pushed into the groove of the spacer ring.
- Push the transflectance sleeve upwards until the spacer disk seats solidly between the transflectance adapter window and the probe window and exactly determines the distance between those two.
- Tighten the retaining screw (1).

NOTE

It is very important that the spacer disk is located on the window center, otherwise it could get stuck between the metal edges during the adjustment procedure, damage the windows and fail to indicate the correct distance between probe and adapter window.



Adjusting the pathlength

NOTE

The socket screw may be replaced by one of the knurled screws for easier exchange.

anymore.

ring by means of the allen key.

adapter from the probe head.

The pathlength of the transflectance adapter is now adjusted to this specific probe head, so that both form a functional unit. The pathlength now matches the engraving of the spacer ring in each case and no additional adjustment is necessary, when you change from one spacer ring to another.

5.7.2 Exchanging the spacer ring

To exchange the spacer ring in order to work with another pathlength, proceed as follows:



Exchanging the spacer ring

• Loosen the screw on the spacer ring by means of the allen key.

Now turn the adjustment ring upwards until there is no clearance between the adjustment ring and the spacer ring

Tighten one of the three socket screws on the adjustment

• Loosen the retaining screw and remove the transflectance

ment ring and the socket screw on the spacer ring.

Now tighten the remaining two socket screws on the adjust-

- Remove the spacer ring.
- Take the new spacer ring you want to operate with, slide it over the transflectance adapter and tighten the screw.

5.7.3 Installing the transflectance adapter to perform measurements

To perform a measurement, slide the transflectance adapter over the probe head and orient it so that the pin at the probe head is located in the groove of the spacer ring. Tighten the retaining screw.

NOTE

When performing measurements, make sure that there are no air bubbles in the measuring gap (pathlength) between probe and adapter window by carefully moving the probe head in the sample.

When you do not perform any measurements with the transflectance adapter, store it in the plastic box.

5.8 Installing measurement equipment to the NIRFlex Fiber Optic SMA



To install the instrument, proceed as follows:

To mount the SMA-adapter onto the optical fiber cable:

• Put the spring onto the fiber connector and secure the end of the connector with the threadened spacer nut.

NOTE

The spring must be freely movable after installation! Fasten the spacer nut handtight only!

- Insert the optical fiber cable.
- Place it into the cable-slot of the stress relief and swivel the stress relief to match the screw heads at the SMA socket.
- Push the stress relief against the spring of the optical fiber cable and swivel it into vertical position.
- Fasten the two fixating screws of the stress relief hand-tight.

• Fixate the optical cable with cable ties.

5.9 Starting up the system

An automatic initialization process starts as soon as the NIRFlex N-500 and the PC are started up. The main components of the spectrometer are identified and its basic functions are checked during that initialization process, e.g. lamp unit, standard wheel, laser unit, measuring cell. The time needed for initialization is about 60 seconds.

5.9.1 System Suitability Test

The **S**ystem **S**uitability **T**est (SST) serves as an internal functionality and performance test of the spectrometer. It is recommended to run an SST after each restart of the NIRFlex N-500 to monitor and verify the system performance.

The following SST values are tested:

- Internal system temperatures (e.g. Laser, interferometer, detectors)
- Linearity
- Signal-to-noise ratio
- Wavenumber stability

At the end of each SST a report protocol will be generated. The test results are compared to values ex factory and inform the user about the system condition.

► The report data are stored in the internal database for service and quality control.

NOTE

The time between two SST requests can be set with the NIRWare Application Designer. The recommended standard value between two tests is 24 hours, a test-run takes approximately 5 min. Test results can be exported and stored in XML-format, e.g. for service purpose.

If the instrument is used in continuous operation (e.g. several days of ON-time), a longer checking interval might be suitable. However, frequent tests should be performed to minimize the risk of erroneous measurement.

5.9.2 Temperature tests in detail

The system is equipped with numerous temperature sensors. Individual lower and upper temperature limits are predefined for each sensor.

→ To pass this test, all actual temperature values must be within the limits.

NOTE

Mind the ambient temperature data as listed in section 3, Technical data.

- If temperature is too low \rightarrow let system warm up sufficiently

5.9.3 NIR linearity test

With this test the linearity of the intensity scale of the spectrometer is being tested. The absorbance of a set of five gray filters in the standard wheel is measured sequentially and compared with reference values.

→ To pass this test, all absorbance factors must be within the tolerance.

5.9.4 Signal-to-noise ratio determination

With this test the ratio of signal power to noise power is determined. A high flux (maximum intensity) and a low flux (10% intensity) beam is being analyzed for a number of wavenumber intervals.

➡ To pass these tests, the average and the minimum values must be within the tolerance.

5.9.5 Wavenumber stability test

The wavenumber stability of the system is checked with the PMMA reference plate of the reference wheel. One absorption peak of the transmission spectrum is being analysed and compared to its reference value.

→ To pass this test, the wavenumber accuracy must be within the tolerance.

5.9.6 SST with NIRFlex Solids Transmittance

The SST for the NIRFlex Solids Transmittance is extended in comparison to the SST for the other measuring cells. It is performed with the special SST sample plate, which is provided with every NIRFlex Solids Transmittance, and the internal standard wheel.

Wavelength accuracy is measured in transmittance using a mixture of Rare Earth Oxides. Noise measurements are performed with open beam and with the 5 mm white standard disk both high and low flux.

The linearity tests are performed for open beam, 1 mm and 5 mm white standard disks together with the grey filters of the internal standard wheel covering the complete photometric dynamic range.

5.10 Reference measurements

Frequent reference measurements compensate for environmental effects, which might influence the spectra. Each measuring cell offers a possibility for the reference measurement, the **External Reference**. Some measuring cells additionally offer the possibility of an **internal referencing**.

External Reference

In order to measure the transmittance or the reflectance of a sample, the measured intensity spectrum of the sample is divided by a reference spectrum. This reference spectrum is either recorded without a sample (in case of transmittance) or with a white standard in the sample position (reflectance). We call this an External Reference. An External Reference is always required.

Internal reference

While the acquisition of an External Reference can be automated in some cases (NIRFlex Liquids, NIRFlex Solids Transmittance, Vial and Tablet add-on for NIRFlex Solids), in other cases the measurement of an External Reference requires user interaction (petri dish and XL add-on for NIRFlex Solids, NIRFlex Fiber Optic Solids, and NIRFlex Fiber Optic Liquids). In these cases internal referencing is used to minimize the need for external referencing and user interaction.

Internal and External Reference measurements provided by the measuring cells/add-ons		
Measuring cell / Add-on	Internal reference	External Reference
NIRFlex Solids with petri dish add-on	Х	manually
NIRFlex Solids with vial add-on	-	built-in
NIRFlex Solids with tablet add-on	-	built-in
NIRFlex Solids with XL add-on	-	manually
NIRFlex Solids with Flow Cell add-on	-	manually

Internal and External Reference measurements provided by the measuring cells/add-ons		
Measuring cell / Add-on	Internal reference	External Reference
NIRFlex Solids with Spinner add-on	-	manually
NIRFlex Solids Transmittance	-	built-in
NIRFlex Fiber Optic Solids	Х	manually
NIRFlex Fiber Optic Liquids	Х	manually
NIRFlex Liquids	-	built-in
NIRFlex Fiber Optic SMA	Х	manually
(+ individual accessories)		

Internal and External Reference measurements provided by the measuring cells/add-ons

without internal reference:

$$Spectrum = \frac{sample}{ext. reference}$$

with internal reference:

$$Spectrum = \frac{sample (recent) / int. reference (recent)}{ext. reference (old) / int. reference (old)}$$

For more information on the reference measurements, see also section 6.

6 Operation

This section describes how to start a measurement and how to prepare a reference measurement and gives recommendations on possible applications of the instrument.

	NOTICE
/!⊁∖	Risk of instrument damage at use with engaged transportation lock.
	Disengage the transportation lock prior use
	Keep shocks and vibrations away

6.1 General recommendations for measuring solids

The calibration wavelength range with a standard fiber optic probe goes from 4500 to 10000 cm⁻¹. For quantitative applications the fiber optic probe should be fixed. Hygroscopic samples show temperature dependency. The samples should either be measured at a constant temperature or the temperature influences must be considered during calibration. Coarse grained samples must be possibly milled before carrying out the measurement. Number of scans: Qualitative calibration: 4–16 (max. 32); quantitative calibration: 16–32; granules: 64. The spectral range of the NIRFlex Solids Transmittance is limited to 6000 cm⁻¹. The number of scans should be 64 by default.

General recommendations f	or measuring solids		
Sampling option	Substance	External Reference measurement	Comment
NIRFlex Fiber Optic Solids	Homogenous powder	White standard in the	
	(qualitative) solids with flat surface	reference cap	
NIRFlex Solids with petri dish	Powder (quantitative)	White standard in a	Make sure that there is
add-on	granules	refer-ence holder (or	enough sample in the dish so
		from the Standardization	that all the light is reflected
		KIL, II AVAIIADIE	by the sample
NIRFlex Solids with XL add-on	Powder in containers	White standard on XL	Make sure that there is a
	pellets	add-on (or from the	constant bulk density, a
		Stanuaruization Kit, II	constant sample container
		available)	container position
NIRFlex Solids with vial	Powder in small glass	White standard in vial	
add-on	vials	add-on	
NIRFlex Solids with Spinner	Powder (quantitative),	White standard in a refer-	
add-on	granules	ence holder	
NIRFlex Solids Transmittance	tablets, capsules,	Open beam (reference	
	powders in small	position in sample plate)	
	plastic cups		

6.2 General recommendations for measuring liquids

For sample measurements with the NIRFlex Liquids, make sure that the cuvettes are placed in the direction of the centre of the cuvette holder. This is important to ensure optimal heat transfer to the sample.

The calibration wavelength range with glass cuvettes goes from 4000 to 10000 cm⁻¹.

The useable wavelength range for measurements with a fiber optic probe is 4500 – 10000 cm⁻¹. Temperature differences can cause a spectral shift. The samples should either be measured at a constant temperature or the temperature influences must be considered during calibration. Avoid air bubbles.

Number of scans: Qualitative calibration: 4–10; quantitative calibration: 16–32 (max 64)

General recommendation for measured	suring liquids		
Sampling option	Substance	External Reference measurement	Comment
NIRFlex Liquids	Transparent liquids	Built-in External Reference (air)	1, 2, 5, 10 mm cuvette with spacer
NIRFlex Fiber Optic Liquids	Transparent liquids	Clean probe head	Max. temperature
	aggressive, trans-		150 °C
	parent liquids		avoid air bubbles
NIRFlex Solids with petri dish add-on and transflectance cover	Clear liquids, turbid liquids	Empty petri dish with transflectance cover	Avoid air bubbles

NIRFlex Solids with flow-cell adapter add-on	Clear liquids, turbid liquids	Clean flow cell	Avoid air bubbles
NIRFlex Solids with Spinner add-on and transflectance cover	Clear liquids, turbid liquids	Empty cuvette with trans- flectance cover	Avoid air bubbles
NIRFlex Fiber Optic Solids with transflectance adapter	Clear liquids, turbid liquids	Clean probe head	Make sure there are no air bubbles in the gap

6.3 Starting a measurement

The spectrometer needs a warm-up time of approx. 15 min. The lamp and the laser within the instrument will be switched on as soon as the NIRWare software is running.

When the instrument has warmed up and the sample is presented to the instrument, you can start the measurement.

Generally, you have two possibilities to do so:

- 1. Press the START button at the measuring cell.
- 2. Click the Start icon (green arrow) in the NIRWare Operator.

NOTE

When you are working with the NIRFlex Fiber Optic Solids or NIRFlex Fiber Optic Liquids probe, you can also use the remote button on the handle to start a measurement.

6.4 NIRFlex Solids

6.4.1 Reference measurement for the Petri dish add-on

(Equivalent procedure for the Standardization kit)

A pop-up will remind the operator to measure the External Reference as soon as a certain time window (predefined in the Application Designer) has passed.

A new External Reference is mandatory

- when a new application is selected
- when an add-on or a new measuring cell is selected
- every time the software is restarted

To measure the External Reference, proceed as follows:

- Remove any sample from the add-on and ensure that the glass window is clean.
- Make sure that the bottom side of the External Reference and its holder is clean.
- Place the holder with the External Reference on the petri dish add-on and measure the External Reference.

6.4.2 Reference measurement for the XL add-on

(Equivalent procedure for the Standardization kit)

A pop-up window will remind the operator to measure the External Reference as soon as a certain time window (predefined in the Application Designer) has passed.

A new External Reference is mandatory

- when a new application is selected,
- when an add-on or a new measuring cell is selected, or
- every time the software is restarted.

To measure the External Reference, proceed as follows:

- Remove any sample from the add-on and ensure that the sample glass window is clean.
- Make sure that the bottom side of the External Reference and is clean.
- Place the External Reference on the XL add-on, rotate it until the mark on the reference is next to the hole in the XL add-on, and measure the External Reference.

6.5 NIRFlex Solids Transmittance

Diffuse transmission measurements are very demanding applications, because only a very small part of the incident light is transmitted. Therefore care must be taken for good signal-to-noise ratio. The design of the NIRFlex Solids Transmittance and its optimized detector ensure best performance. NIRFlex Solids Transmittance uses a low energy source, which avoids any overheating of the samples and therefore enables the analysis of temperature sensitive active ingredients. Emissions e.g. from fluorescent tubes can cause artifacts in the sample spectrum. Therefore the cover of NIRFlex Solids Transmittance must be closed during measurements.



NOTICE

Risk of incorrect measurement results by ambient light

Keep the top cover closed during a measurement

Typical samples for the NIRFlex Solids Transmittance include tablets, hard gelatin capsules, powders or soft gelatin capsules including semi-liquids or oily liquids, which requires a very high dynamic photometric range of the system. The design of the measuring cell takes this requirement into account. Without any special settings the user will generate spectra with optimized signal-to-noise ratio for his specific samples.

For reproducible and accurate results in tablet transmission measurements, it is essential to prevent stray light caused by light passing around the side of the tablets. Therefore the sample plates are individually customized in order to fit perfectly for your tablets and to ensure the optimal performance. Additionally the sample plates exhibit a specially selected coating to prevent reflection and light leakage.

All sample plates include an External Reference position. By default reference measurements are performed automatically before every measurement sequence.



NOTICE

Risk of damage at the coating of the sample plate

• Do not cleat the sample plate in a dishwasher

Clean the sample plate with Ethanol and a soft, lint-free tissue

6.6 NIRFlex Liquids

System preparation

•

The liquid cell must reach a stable working temperature before a measurement can be performed. The heat-up time depends on the ambient temperature.

Starting at standard ambient temperature of 25 °C the measurement cell needs:

- approximately 15 minutes to stabilize at 35 °C.
- approximately 30 minutes to stabilize at 60 65 °C.

NOTICE
Risk of measurement errors by unsufficient heat-up time
Cuvettes and sample material must be completely heated-up at measurement

Depending on the heat distribution and the positioning of the cuvettes the temperature difference between probe and liquid temperature is about 2 °C at a measurement temperature of 65 °C.





NIRFlex Liquids measurement cell



CAUTION

A

Minor or medium burns by hot surfaces

Do not touch the cell chambers

NOTE

The software does not prevent measurements in case of incorrect temperatures. The operator is responsible, that the current temperature corresponds with the set temperature. A reproducible temperature of the samples is crucial when carrying out measurements in transmission mode. The heat-up time of the sample material must be sufficient to reach the measurement cell temperature.

Working with different types of cuvettes

For most of the applications running with the NIRFlex Liquids, the use of 2 mm cuvettes is recommended. However, the cell is designed to use 1, 5 and 10 mm cuvettes as well. If doing so, the spacers, which fix the cuvettes in the measurement position, must be changed. A set of different spacers is included in the scope of delivery.



Handling of different cuvette types

For quadratic cuvettes : No spacers are required.

For rectangular cuvettes:

All rectangular cuvette types must be installed in pairs with a matching spacer!

- The cuvette must be aligned to the turning hub (inner circle of the cell).
- A matching spacer must be aligned to the outer circle of the cell.

NOTE

Quartz glass cuvettes are recommended when working with the NIRFlex Liquids. Most plastic cuvettes show a significantly higher light absorption rate which can reduce the determination performance. This also has direct influence onto calibration and reproducibility. Make sure that the radiated surfaces of the cuvettes are placed perpendicular to the beam of light.



WARNING

A

Risk of fire by highly flammable materials

Do not insert highly flammable materials or samples into the measurment cell

6.7 NIRFlex Fiber Optics

All fiber optics must be treated with special care to avoid breaks and other damages of the glass fibers and optical surfaces! Cleaning should always be done manually with a soft, lint free damp cloth with mild soapy water. Optical surfaces can be cleaned with Kimwipes.

	NOTICE
¥!	Risk of damage at the fiber optics
	Do not bend the fiber optics to a radius smaller than 15 cm
	Do not overstrech or twist the fiber optics
	Do not expose the fiber optics to heavy impacts or punctual pressure
	• Do not bring the fiber optics into contact with organic solvents, acids or alkaline solutions
	Do not bring the white standard into contact with furanes, acids and other oxidizing agents

6.7.1 NIRFlex Fiber Optic Solids

The handle is equipped with a remote control for initiating the measurements remotely and displaying the results using LEDs on the handle.

Because the fiber optic probe is used at a certain distance from the spectrometer, the length of the fiber optic cables between the measuring cell (spectrometer) and the probe tip is about 2 m (special versions with longer fiber optic cables are available on request).



NOTE

- The software automatically detects the presence of a transflectance adapter but not the pathlength in use, so make sure to use the appropriate pathlength.
- It is not possible to start a reference measurement if the reference cap has not been detected by the system.

Reference measurement

For correct measurements and correct results, it is necessary to record a reference spectrum regularly. For this purpose, use the reference cap. In order to avoid false results, the reference cap is detected automatically if installed.

6.7.2 NIRFlex Fiber Optic Liquids

For most of the applications a pathlength of 1 or 2 mm is the best choice. Many samples will contain water or alcohol, which cause strong NIR absorptions. Therefore for the standard NIRFlex Fiber Optic Liquids a pathlength of 2 mm will be sufficient (special versions with other pathlengths are available). For sample measurements with the NIRFlex Fiber Optic Liquids make sure that the quartz window in the probe head is in full contact with the sample to be analyzed.

Despite its rugged design, the transmission probe requires careful treatment.

6.7.3 NIRFlex Fiber Optic SMA

This measurement setup is suitable for continuous online measurements. Special settings can be found in the NIRWare manual.

NOTE

Respect handling and operation information provided by the manufacturer of your custom optics / accessories.

7 Maintenance

This section gives instructions on all maintenance work to be performed in order to keep the instrument in good working condition. In addition to this, adjustment jobs the operator can carry out by himself/herself are explained.

	A CAUTION
	Risk of dangerous device status or defects and malfunctions when serviced by untrained personnel
	 Service and maintenance actions which require to open the device housing must be performed by trained personnel only
<u>5555</u>	• Switch off the device and disconnect the mains cable before any service, maintenance or cleaning
	Do not touch live parts
	Close the housing before reconnecting the device to mains

7.1 Cleaning

To avoid cross-contamination and germ dissemination (e.g. by bacteria) the operator is advised to attend common laboratory biosafety measures in accordance the chosen application and environment. The "Laboratory Biosafety Manual" of the World Health Organization can be used as a practical guidance.

WARNING
Risk of cross-contamination and germ dissemination at use
Exchange filter regularly
Keep the housing and the working area clean
Keep all surfaces in contact with sample material clean and free of germs
Use disinfectant to avoid germ dissemination
Do not reuse sample material

7.2 Housing

Clean the housing with a soft, lint free damp cloth with mild soapy water. For hygienic reasons, use surface disinfectant wipes in combination with similar disinfectant spray on alcohol basis. If the sample window on top of the instrument is dirty, clean it with an antimicrobial wipe (e.g. Melisptol® wipes).

	NOTICE
!	Risk of damage at the housing
	Do not use any objects for cleaning which might scratch the optical surfaces or add-ons
	Do not clean coated optical surfaces in a washer.

In order to avoid contamination by sample material between the measurements, clean the surfaces in direct contact with the sample after each measurement.

NOTE

- Switch off the NIRFlex N-500 and all directly connected eletronic devices i.e. monitors before cleaning the housing.
- Avoid scratches on optical surfaces as they can influence a measurement!
- For coated optical surfaces, use abrasion-resistant precision cloths such as Kimwipes from Kimberly-Clark. Primary cleaning can be done with disinfectant wipes on alcohol basis.
- Avoid fingerprints on any optical surface!

7.2.1 Optical surfaces and probes

All optical surfaces must be handled with care i.e. must not be scrached, cleaned with unproper cleaning medium. Do not apply excessive force to any part of the device or its accessories!

If the connectors for the measuring cell on the instrument are dirty, clean them with a soft cloth moistened with alcohol.

In order to avoid contamination by sample material between the measurements, clean the surfaces in direct contact with the sample after each measurement. For this purpose, use abrasion-resistant precision cloths such as Kimwipes from Kimberly-Clark. Loose dust can be removed by blowing with **clean** and **oil-free** compressed air. If substances still adhere, you may additionally rinse the surfaces with acetone or alcohol.

Due to the limited resistance of the adhesive between metal and quartz glass, the fiber optic probe must not be in prolonged direct contact with organic solvents nor acids and alkaline solutions. Take it out of the liquid directly after completing the scans.

To prevent contamination of the white standard reference make sure that it is always applied to a clean measuring cell.



7.2.2 Cleaning of the External Reference

For dependable reference measurements the External Reference and Transflectance adapter must be clean and damage free. In case the External Reference is strongly contaminated despite the necessary precautions, rinse it with pure acetone. Make sure to remove all cleaning residue.



	NOTICE
! }	Risk of damage at the External Reference
	Do not drop the External Reference
	Do not scratch the surface
	Never bring it in contact with hard objects.
	• Do not use any cleaning media with abrasive character which might scratch the surface.
	• Do not use item for reference measurement when damaged or scratched.
	• Do not use any compressed air for blowing off dirt from the External Reference, as it often contains oil and is not sufficiently pure.

After treating the External Reference with pure acetone, always make sure that the acetone adsorbed by the reference material evaporates completely.

For this purpose, you can place the External Reference in an oven for a few minutes at a maximum of 60 °C.

The reference material adsorbs oils and fats.

NOTE:

The same cleaning procedure could be carried out for the standardization kit.

7.2.3 Cleaning of the Transflectance adapter

The transflectance adapter should be cleaned regularly after each measurement in order to avoid a sample cross contamination during the next measurement.

To clean the transflectance adapter you can rinse it with solvents suited for use with the materials of the adapter and probe (stainless steel, quartz glass, fluorine rubber). After rinsing, dry the adapter thoroughly and carefully inspect it for remaining contaminations.

Suited cleaning solvents are water, acetone, and alcohols.



NOTE

- Exact cleaning procedures tailored to the individual sample composition have to be developed by the customer.
- When you analyze liquids of medium or high viscosity, sample residues may accumulate in the measuring gap and all other gaps between the adapter and the probe. In this case, remove the transflectance adapter from the probe head to clean it thoroughly in order to avoid a sample cross-contamination during the next measurement.

7.3 Advanced testing with NIRWare Automatic Diagnose

In addition to the SST NIRWare 1.x includes the NIRWare Automatic Diagnose (NADIA). NADIA is a tool which enables Buchi service personnel to judge the condition of the NIRFlex N-500 or to do a performance check of the instrument as part of a service contract.

```
To start NADIA select Start > Programs > Buchi NIRSolutions > NIRWare > Nadia
```

Close the NIRWare Operator before starting NADIA. The Control System Service must be stopped during the test. NADIA will prompt you for permission to stop the Control System Service, if it detects a running Control System Service.

Click '**Start**' to execute the Nadia test and follow the instructions on the screen. Once the test is finished, the following information is displayed:

	🗄 NIRware Automatic Diagnose (Nadia)		- 0×
	[12-01-2007 09:10:02.591] NADIA	- NIRWare Automatic Diagnose (Nadia)	~
	[12-01-2007 09:10:02.601] Versio	on: 10.2.2.0	
	[12-01-2007 09:10:02.611] Starte	ed at 12.01.2007 09:10:02	
	[12-01-2007 09:10:02.821] eMessa	ageLog: eInfo The ControlSystem is still running! May I stop the	Control
	[12-01-2007 09:11:49.264] Start	check Embedded system	
	[12-01-2007 09:11:49.605] Start	check Embedded Parameters	
	[12-01-2007 09:11:50.666] Start	check Integrity Instrument	
	[12-01-2007 09:12:01.111] Start	check Instrument	
	[12-01-2007 09:14:28.423] Start	check Measurement Cell	
	[12-01-2007 09:15:52.684] try to) start the subsystem IU	
	[12-01-2007 09:16:08.487] try to) start the subsystem MCST	
	[12-01-2007 09:16:17.500] Finish	ned	
	Datafile "C:\Documents and Setti	ings\kelo\My Documents\MyNadiaFiles\LL-2007-01-12_09-16-17.xml"	created
	Nadia Run finished		
	<		
ŀ	<u>)</u>		
	Current		E-a 1
	Didit		

Test result Nadia

The result is saved in an *.xml file, which is stored in the folder shown below:



MyNadiaFiles

Send this file to your customer service organisation for evaluation. To archive the file, copy it to a different folder.

7.4 Lamp module replacement

The primary lamp module needs to be replaced quickly after failure. This action can be performed by a trained operator.

After failure of the primary lamp module, the following instructive message appears:

Message:OP.121	\sim
0	The secondary lamp is in use. It is recommended to replace the primary lamp. If you already replaced the primary lamp, activate it as follows: - Open service tools - Go to Assembly Exchange, then Lamp - Click the Button "Active Primary Lamp" - Reset the Primary Lamp Counter
9	

NOTE

When the primary lamp is broken the instrument automatically switches to the secondary lamp. In this case make sure to replace the primary lamp as soon as possible as the instrument does not work anymore in case of failure of the secondary lamp and this lamp has to be replaced by a Buchi service engineer.



To replace a defective lamp module, proceed as follows:



- Switch off the instrument and unplug the mains cable. Let it cool down safely!
- Loosen the two screws on the left rear of the instrument approximately 3-5 turns.
- Remove the cover.

For easy release of the housing slightly tilt the back part away from the screws. Then pull out the housing towards the rear side of the instrument. There is a built-in sensor to record the opening of the housing. A safety tack ensures that the lamp remains in the correct position.

- Slide the safety tack to the upright position (counterclockwise).
- Pull the metallic housing with the primary lamp out of the lamp unit.
- Exchange the primary lamp source with a new lamp by removing it from the gray plug and connect the new primary lamp source to this plug.



 Insert the new lamp into the lamp unit and slide the safety tack back to the horizontal position until a metallic click sound is audible, which indicates the right position. Make sure that the gray plastic plug is in a vertical position.



Replacing the lamp module

Secure the housing of the lamp unit with the two screws on the back side of the unit.



NOTICE

Risk of instrument damage by ingress of particles.

- Check proper seating of all housing parts before use
 - Do not operate instrument when housing is open or unlocked

Resetting the lamp lifetime counter

•

After lamp module exchange, the following message appears at power on:



Message concerning required instrument setup

- Click the OK icon *solution* to confirm the message.
- Now click the Service icon 🥩 (Operator) and double-click 'Assembly'.

NOTE

To correctly monitor the lamp lifetime the software hour counter has to be reset after each lamp replacement. At lamp exchange, the secondary lamp module is active.

Assembly Setup Assembly Article State Access Main Board 46134 done denied æ 1 42725 Interferometer done denied æ 12 Detector Board 45360 done denied æ M Standard Wheel 46119 done denied æ M Lamp 42774 open granted æ m Solids 46212 denied æ M done Laser Module 42787 done granted æ M Application Board 45365 done denied æ M Cell Detector Unit 45323 done denied æ 1

The following dialog opens:

Assembly setup dialog

• Click the icon next to the Lamp row 🖻 to reset the "housing open" status.

Workflow to reset the lifetime counter of the primary lamp module

To establish the standard working conditions of the NIRFlex N-500:

NTWest Operator	
	иент
Control State Control State Control State	
Sandy Endown - 42 Emmi - 42 Emmi Primery Lamp Incurs of operation 5488	
- @ calcular Paranter was secondary Lamp hours of operation 14	
Aduation: The secondary lamp is active	
	Attention: The secondary lamp is active
	te Primary
48	
Constant	00000
	Primary Lamp hours of operation 5480
	m
•	\sim
•	Message:IS.1022
NEWww.Dpenter	
	Do you really want to reset the lifetime counter 'Primary Lamp'?
Verent Conjugator Verent Conjugator Verent Conjugator Verent Conjugator Verent Conjugator Verent Ve	
assets Endrage - 4 Emmi - 4 Emmi - 4 Emmi - 4 Emmi Primary Lamp hours of operation 0	
- g2 Colification Francedor which Secondary Lamp hours of operation () branced	
<i>i</i>	
Uperator Sector Task Operator IP State S	MADUT States

- Start the Operator software
- Switch from "secondary lamp" (1) to "primary lamp"
- Resetting the hour ② counter will bring up a prompt for confirmation
- Confirm the message (3) to finally set the lamp lifetime counter to zero
- → After this procedure, reboot the NIRFlex N-500 and restart the Operator software.
7.5 Laser unit replacement

The LASER unit needs to be replaced at failure. This action can be performed by a trained operator or (preferably) by a service technician.





White plug at laser power supply



To replace the laser, proceed as follows:



- Loosen the two screws on the left rear of the instrument approximately 3-5 turns.
- Remove the cover.

For easy release of the laser housing it is recommended to slightly tilt the housing and then pull it horizontally away from the instrument. There is a built-in sensor to record the opening of the housing.





Removing the LASER unit

• Disconnect the electric lasers power supply plug.

- Loosen the two screws which are on the laser unit bottom plate.
 For easy release of the laser it is recommended to lift it upright and than pull the laser unit backwards.
- Now take the new laser unit, insert it and make sure that its base plate is correctly placed into the bolts.
- Fix the new laser unit with the two screws and connect the electric plug.
- Close the housing of the laser unit. This can easily be done by first positioning the slits over the screws.

When the instrument and the PC is switched on again and the NIRWare Operator is started, a corresponding message will be displayed and inform you that this change in hardware configuration must be added into the database.

For installation proceed in reverse order.

	NOTICE
!!	Risk of instrument damage by ingress of particles.
	Check proper seating of all housing parts before use
	Do not operate instrument when housing is open or unlocked

Resetting the laser lifetime counter

After LASER unit replacement the following message appears:



Message concerning required instrument setup

- Click the OK icon white to confirm the message.
- Now click the Service icon 🧟 (Operator).

The following dialog opens:

	Assembly Setup					
	Assembly	Article	State	Access		
1	Lamp	42774	done	granted	산	M:
2	Laser Module	42787	open	granted	장	M
3	Interferometer	42725	done	denied	橙	M
4	Standard Wheel	46119	done	denied	산	M
5	Main Board	46134	done	denied	æ	M

Assembly setup dialog

Excl	nange Sequence:Laser Module
Laser hours of operation	1064

Exchange sequence dialog for the laser

Activate the watch icon next to the line Laser hours of operation

The following message box is displayed:

Message:I5.1022		×
?	Do you really want to reset the lifetime counter 'Laser'?	

Message

Confirm the message by clicking the OK icon

The following screen indicates the laser lifetime counter is set to zero:



Hours of operation of the laser

• Now click the Go Back icon 5 to finish the procedure.

7.6 Filter pad replacement



When the filter pads at the rear of the instrument and the measuring cell are dirty, ventilation and thermal stability of the instrument will no longer be ensured.

Replace the pad after 6 month of use at the latest. An earlier exchange might be necessary, when the instrument is operated in a dusty environment.



To replace the ventilation filter, proceed as follows:

- Remove the filter cover.
- Remove the old filter pad.
- Insert the new filter pad.
- Reinstall the filter cover.

Replacing the filter pads

	NOTICE
! }	Risk of instrument damage by ingress of particles.
	Check proper seating of the filter and filter bracket before use
	Do not operate instrument without filter
	NOTICE
! }	Risk of instrument damage by overheating.
	Do not block the cooling openings of the instrument
1	

7.7 Main fuse replacement

The primary fuses are located at the rear of the instrument next to the power supply socket. They are accessible from the outside.

To replace the primary fuses, proceed as follows:



- Unplug the power cord!
- Remove the defective fuses from the holder and replace them with new fuses of the same rating (T 10 A, L 250 V).
- Reconnect the power supply connector and switch on the instrument.

Replacing the primary fuses

7.7.1 Measuring cell fuse replacement

Each measuring cell is provided with two fuses located at the bottom. To replace them, proceed as follows:



Location of the fuses at the bottom of the measuring cell

- Remove all add-ons, petri dishes, vials, etc. from the measuring cell.
- Remove the measuring cell from the instrument.
- Turn the cell, in a way that you see the bottom side. The two black fuse holders are located as indicated by the arrow on the picture to the left.
- Replace the 2.0 AT fuses with two new ones.

7.8 NIRFlex Solids

It is possible that solids or pasty samples are spilled into the sample holder of the NIRFlex Solids. In this case it is recommended to start the cleaning process as soon as possible to prevent any damage of the instrument. Special attention is needed on that point, if aggressive media are spilled over the instrument.

To clean the measuring cell, proceed as follows:

- Remove the add-on from the measuring cell.
- If some solid residues are left in its sample holder, remove the cell from the instrument and turn it, so that the residues fall off.
- If some pasty residues are left in the sample holder, remove them with a soft tissue and clean the sample holder with a non-aggressive liquid (water or alcohol).

Special attention is needed to prevent the glass window from getting any scratches by using optical tissues.

7.9 NIRFlex Liquids

To clean the measuring cell, proceed as follows:

- Take the measuring cell out of the basic instrument.
- Remove the cuvettes that might still be in the openings.
- Remove the sample holder from the measuring cell by loosening the screws in sample position 1, 3 and 5.
- Clean the sample holder and the corresponding opening of the measuring cell. Make sure that there are no residuals of sample anymore.
- Place the sample holder back into the measuring cell and retighten the three screws.





NIRFlex Liquids measurement cell

7.10 Customer service

Only authorised service personnel is allowed to perform repair work at the instrument. These persons have a comprehensive technical training and knowledge of possible dangers which might arise from the instrument.

Addresses of official Buchi customer service offices are given on the Buchi website under: www.buchi.com. If malfunctions occur at your instrument or you have technical questions or application problems, contact one of these offices.

The customer service offers the following:

- Spare part delivery
- Repairs
- Technical advice

8 Storage, transport and disposal

This section instructs how to handle the instrument for storage or transport, and gives advice on disposal.

	NOTICE
! ¥	Risk of instrument damage at storage and transport
	Engage the transportation lock before storing, transport and re-packaging
	Only store and transport the instrument in original packaging
	Keep shocks and vibrations away
	Safely store and transport accessories separately

8.1 Storage and transport

The NIRFlex N-500 is equipped with a mechanical transportation lock to avoid damage of the interferometer during transportation and shipping by shocks and vibrations. For storage and transportation, the transportation lock must be in "Locked" position. Manual handling of the instrument requires a second person!

NOTE

- Because of its weight it is recommended to carry the instrument with two persons.
- Clean the instrument thoroughly before storage or transport.
- When returning the instrument to BUCHI fill out a copy of the "Health and Safety Clearance" form and safely attach it at the outside of the transport box.

8.2 Disposal

For instrument disposal in an environmentally friendly manner, a list of materials is given in section 3.1. This helps to ensure that the components can be separated and recycled correctly by a specialist for disposal.

You have to follow valid regional and local laws concerning disposal. For help, please contact your local authorities!

A CAUTION
Risk of poisoning by improper decomposing and recycling of the device
Do not dispose of the device as unsorted household waste
Recycle all materials according to local directives and laws

NOTE

When returning the instrument to the manufacturer for repair work, please copy and complete the health and safety clearance form on the following page and enclose it with the instrument.

Health and Safety Clearance

Declaration concerning safety, potential hazards and safe disposal of waste.

For the safety and health of our staff, laws and regulations regarding the handling of dangerous goods, occupational health and safety regulations, safety at work laws and regulations regarding safe disposal of waste, e.g. chemical waste, chemical residue or solvent, require that this form must be duly completed and signed when equipment or defective parts were delivered to our premises.

Instruments or parts will not be accepted if this declaration is not present.

Equipment

Model:

Part/Instrument no.:

1.A Declaration for non dangerous goods

We assure that the returned equipment

- has not been used in the laboratory and is new
- was not in contact with toxic, corrosive, biologically active, explosive, radioactive or other dangerous matters.
- is free of contamination. The solvents or residues of pumped media have been drained.

1.B Declaration for dangerous goods

List of dangerous substances in contact with the equipment:

Chemical, substance	Danger classification

We assure for the returned equipment that

- all substances, toxic, corrosive, biologically active, explosive, radioactive or dangerous in any way which have pumped or been in contact with the equipment are listed above.
- the equipment has been cleaned, decontaminated, sterilized inside and outside and all inlet and outlet ports of the equipment have been sealed.

2. Final Declaration

We hereby declare that

- we know all about the substances which have been in contact with the equipment and all questions have been answered correctly
- we have taken all measures to prevent any potential risks with the delivered equipment.

Company name or stamp:			
Place, date:			
Name (print), job title (print):			
Signature:			
Health and Safety Clearance_20081110.	doc_20081110	Version 1.0	Page 1/1

9 Spare parts and accessories

This section lists spare parts, accessories and options of the instrument including their ordering information.

Order the spare parts from Buchi. Always state the product designation and the order number when ordering spare parts.

Use only genuine consumables and genuine spare parts for maintenance and repair to assure good system performance and reliability. Any modifications to the spare parts used is allowed only with the prior written permission of the manufacturer.



CAUTION

Risk of device damage and dangerous system status by unauthorized spare parts

Only use original spare parts

9.1 Scope of delivery

9.1.1 Interferometer



Interferometer	
Product	Order number
NIRFlex N-500 Polarization Interferometer	N505-000
100-230 V, 50/60 Hz	

9.1.2 Measuring cells



NIRFlex Solids	
Product	Order number
For measurements in diffuse reflection mode with rotating sample desk	N510-000
Petri dish add-on	N510-001
Vial add-on	N510-002
XL add-on	N510-003
Tablet add-on	N510-004
XL add-on with iris aperture	N510-005
XL add-on accepting B+L sample cups	N510-006
Spinner add-on	N510-012
Flow-cell adapter add-on	N510-013



NIRFlex Solids Transmittance	
Product	Order number
For measurements in diffuse transmission mode with rotating sample plates	N514-000
Sample plate for 30 tablets with a diameter of 4 - 12 mm	N514-002
Sample plate for 10 tablets with a diameter of 12 - 30 mm	N514-001
Sample plate for 10 tablets with iris apertures	N514-003



Table 3-4: NIRFlex Liquids	
Product	Order number
For measurements in transmission mode	N511-000
using cuvettes	

Order number



NIRFlex Fiber Optic Solids	
Product	
For measurements in diffuse reflection	

For measurements in diffuse reflection	
2 m fiber length	N512-000
3 m fiber length	N512-004
5 m fiber length	N512-005



NIRFlex Fiber Optic Liquids	
Product	Order number
For measurements in transmission mode using a fiber optic probe (default length 2 m, custom-made lengths up to 7 m	N513-000
available)	

NIRFlex Fiber Optic SMA		
Product	Order number	
For measurements in transmission or diffuse reflection mode. External optical probes or and measuring cells can be connected through fiber optic via SMA- connector	N515-000	
Monofiber without SMA connectors, 1m	A515-020	
SMA connectors with tailoring	A515-021	
Transmission immersion probe	A510-020	
Transmission flow cell	A510-010	

9.1.3 Software



Software	
Product	Order number
NIRWare Basic	N550-011
NIRWare Advanced	N550-013
NIRCal	N550-021

9.1.4 Standard accessories





NIRWare S Quick Guide



E74



Standard accessories		
Product	Order number	
1 Power supply cable		
Туре СН	10010	
Type Schuko	10016	
Type GB	17835	
Type USA	10020	
Type AUS	17836	
1 Ethernet connection cable NIRFlex	48457	
N-500/PC		

Operation Manual NIRFlex N-500

English	11593575
German	11593576
French	11593577
Italian	11593578
Spanish	11593579

Quick Guide NIRWare

Quick Guide NIRCal with Toolbox

English	11593597
German	11593598
French	11593599
Italian	11593600
Spanish	11593601
Chinese	11593603
Japanese	11593602

9.1.5 Optional accessories











Optional accessories		
Product	Order number	
Set of 2 quartz glass cuvettes with 2 mm	46266	
pathlength for NIRFlex Liquids		

1 spacer for 5 mm cuvettes	45430
1 spacer for 2 mm cuvettes	45431
1 spacer for 1 mm cuvettes	45432
Set of spacers (6 pieces of each)	N560-013

Set of 200 glass vials to be used with	N560-015
NIRFlex Solids with the vial add-on	

Vial adapter for NIRFlex Liquids N511-001 with a diameter of 8 mm, including 100 vials

Glass petri dishes 10 pcs. (up view) 11072073 Not suitable for use with Transflectance Cover

	Ontional appagation (point)	
	Product	Order number
	Transflactance cover or high performance	/1626
	cup using 100 mm petri dishes	41030
	XL add-on for NIRFlex Solids with iris	N510-005
	aperture	
	XL add-on for NIRFlex Solids accepting	N510-006
	B+L cups	
	High performance comple out for NIDEloy	46250
	Solids	40239
	Exchange glass bottom	46246
	Transflectance adapter for NIRFlex Fiber Optic Solids	N512-006
	The transflectance adapter is supplied	
	in a plastic box containing the following parts:	
(4)	① Adjustment ring	
6665	② Spacer rings for pathlength 0.5 mm, 1 mm and 1.5 mm	
	③ 3 knurled screws	
	④ 1 mm spacer disk	
	(5) Allen key	
	(6) Transflectance sleeve	







Optional accessories (cont.)	
Product	Order number
Holder for fiber optic probe	46273
	10210

Barcode reader

42762

Special Vials, set of 6

11056492

Unbreakable Sample Cup for NIRFlex	11055058
Solids	
Transflectance cover for Unbreakable Cup	11055998
Spinner add-on	11055087





Standardization kit

11069861



Optional accessories (cont.)	
Product	Order number
Stamp for Hellma cuvettes	11056459

Flow add-on	11055051
Flow Through cuvettes	11055911

9.2 NIRFlex spectrometer

Spare parts NIRFlex spectrometer	
Product	Order number
Primary lamp module	N560-001
Laser unit	N560-002
Fuse (T 10 A slow blow, L 250 V)	02009
Filter cover and filter pad	N560-004
1 Ethernet connection cable NIRFlex N-500/PC	48457
Power supply cable	33748
10 filter pads	42779

9.3 NIRFlex Solids

Spare parts NIRFlex Solids	
Product	Order number
Fuse 2.0 AT	02998
External Reference	N560-003
Vial add-on	N510-002
XL add-on	N510-003
Petri dish add-on	N510-001
Tablet add-on	N510-004
Spinner add-on	N510-012
Flow-cell adapter add-on	N510-013
Set of 10 glass petri dishes, 10 mm Not suitable for use with Transflectance Cover.	11072073
Set of 200 glass vials for vial add-on	N560-015
10 filter pads	42778
XL add-on with iris aperture	N510-005
XL add-on accepting B+L sample cup	N510-006
High performance sample cup	046259
Exchange glass bottom for high performance sample cup	046246
Transflectance cover	041636
Set of 6 special vials for Vial add-on	11056492
Unbreakable sample cup	11055058
Transflectance cover for Unbreakable sample cup	11055998
Spinner add-on	11055087
Transflectance cover for Hellma cuvette	11056376
Flow cell add-on	11055051
Flow-through cuvette	11055911
Standardization kit	11069861

9.4 NIRFlex Solids Transmittance

Spare parts NIRFlex Solids Transmittance	
Product	Order number
Fuse 2.0 AT	02998
10 filter pads	42778
Window, complete	48961
Tweezers	48959
Cover for NIRFlex Solids Transmittance	48955

9.5 NIRFlex Liquids

Spare parts NIRFlex Liquids	
Product	Order number
Fuse 2.0 AT	02998
Cuvette holder complete	N560-010
Set of 2 quartz glass cuvettes, 2 mm	46266
1 spacer for 5 mm cuvettes	45430
1 spacer for 2 mm cuvettes	45431
1 spacer for 1 mm cuvettes	45432
10 filter pads	42778
Vial adapter for 8 mm vials including 100 vials	N511-001

9.6 NIRFlex Fiber Optic Liquids

Spare parts NIRFlex Fiber Optic Liquids	
Product	Order number
Fuse 2.0 AT	02998
10 filter pads	42778

9.7 NIRFlex Fiber Optic Solids

Spare parts Fiber Optic Solids	
Product	Order number
Fuse 2.0 AT	02998
Sample probe head	48400
10 filter pads	42778

9.8 NIRFlex Fiber Optic SMA

Spare parts Fiber Optic Solids	
Product	Order number
Fuse 2.0 AT	02998
10 filter pads	42778
Packing parts SMA	11056060

9.9 Transflectance adapter

Spare parts transflectance adatper	
Product	Order number
Spacer ring 0.5 mm	48994
Spacer ring 1 mm	48995
Spacer ring 1.5 mm	48996
Knurled screw	44311

10 Declarations and requirements

10.1 FCC requirements (for USA and Canada)

English:

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to both Part 15 of the FCC Rules and the radio interference regulations of the Canadian Department of Communications. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Français:

Cet appareil a été testé et s'est avéré conforme aux limites prévues pour les appareils numériques de classe A et à la partie 15 des réglementations FCC ainsi qu'à la réglementation des interférences radio du Canadian Department of Communications. Ces limites sont destinées à fournir une protection adéquate contre les interférences néfastes lorsque l'appareil est utilisé dans un environnement commercial.

Cet appareil génère, utilise et peut irradier une énergie à fréquence radioélectrique, il est en outre susceptible d'engendrer des interférences avec les communications radio, s'il n'est pas installé et utilisé conformément aux instructions du mode d'emploi. L'utilisation de cet appareil dans les zones résidentielles peut causer des interférences néfastes, auquel cas l'exploitant sera amené à prendre les dispositions utiles pour palier aux interférences à ses propres frais.

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