OPUS

Spectroscopy Software

Version 6

User Manual

STEP

BRUKER

Table of Contents

1   Introduction

The OPUS/STEP software package allows a wide range of kinetic and modulation/demodulation experiments based on the step-scan technique. In contrast to the normal Rapid Scan mode of data acquisition with an uniform interferometer mirror speed, in the Step Scan mode the interferometer mirror is moved from one optical retardation position to the next in a stepwise manner and data is acquired at each position.

Typical applications of the Step-Scan technology are:

- Photoacoustic spectroscopy (PAS) measurements with variable modulation frequencies (depth-profiling) and photothermal beam deflection,
- Modulation/demodulation experiments where the dynamic spectral response of the sample to a perturbation is determined,
- Time-resolved spectroscopy (TRS) to pursue rapid dynamic phenomena into the nanosecond domain.

Time resolved step scan measurements can be performed either with the internal analog-to-digital converter or a transient recorder. The results of a time-resolved Step Scan experiment are stored in a 3D file. To display or process these files the OPUS/3D software package is required.

2   Hardware Requirements

Computer System

The following requirements must be met to ensure an optimum operation the of OPUS/STEP Software:

- one ISA slot for the Acquisition Processor (AQP)
- depending on the application (e.g. for time resolved measurements in the nanosecond domain), a second ISA slot is required

Note: PC data stations that fulfill the above listed requirements are available directly from Bruker.

Spectrometer

The Step Scan option is available on the following Bruker FT-IR spectrometers:

- EQUINOX 55
- IFS 66/S
- IFS 66v/S

To perform step scan measurements, hardware and firmware upgrades (i.e. step scan option, part number S 510) of the base configuration of the above listed spectrometers are required.

> **Note:** The OPUS/STEP software package is included in the step scan option, part number S 510.

For particular applications additional options and/or accessories might be necessary. For further information contact your local Bruker representative.

3   Step Scan Modulation

3.1   General Information

This measurement mode is designed for photoacoustic spectroscopy (PAS) and other modulation/demodulation experiments. First, the IR beam is modulated at one or more discrete frequencies. Then, the detector signal is demodulated at the same frequency, so that the intensity at a given frequency and phase angle (relative to the modulation signal) can be measured and quickly determined for every interferometer mirror position.

There are two kinds of modulation: amplitude modulation (e.g. by chopping the IR beam or by manipulating the sample itself periodically, such as stretching or photoelastic modulating) and phase modulation (i.e. periodic vibration of the interferogram mirror about each set position). The demodulation is performed either by an external lock-in amplifier (LIA) or, if the DSP option is available, by an internal digital signal processor (DSP).

3.2   Setting up Measurement Parameters

Select in the OPUS *Measure* menu the *Step Scan Modulation* function. The *Step Scan Modulation* dialog box opens. Basically, this dialog box is identical to the *Measurement* dialog box (described in the OPUS Reference Manual), except for the *Step Scan Modulation* page.

Click on the *Step Scan Modulation* tab. The dialog window shown in figure 1 opens. It allows you to define the necessary parameters to perform a step scan modulation measurement.

> **Note:** Before starting a measurement ensure that **all** parameters are set correctly. For detailed information on the measurement parameters of the other dialog windows refer to the OPUS Reference Manual.
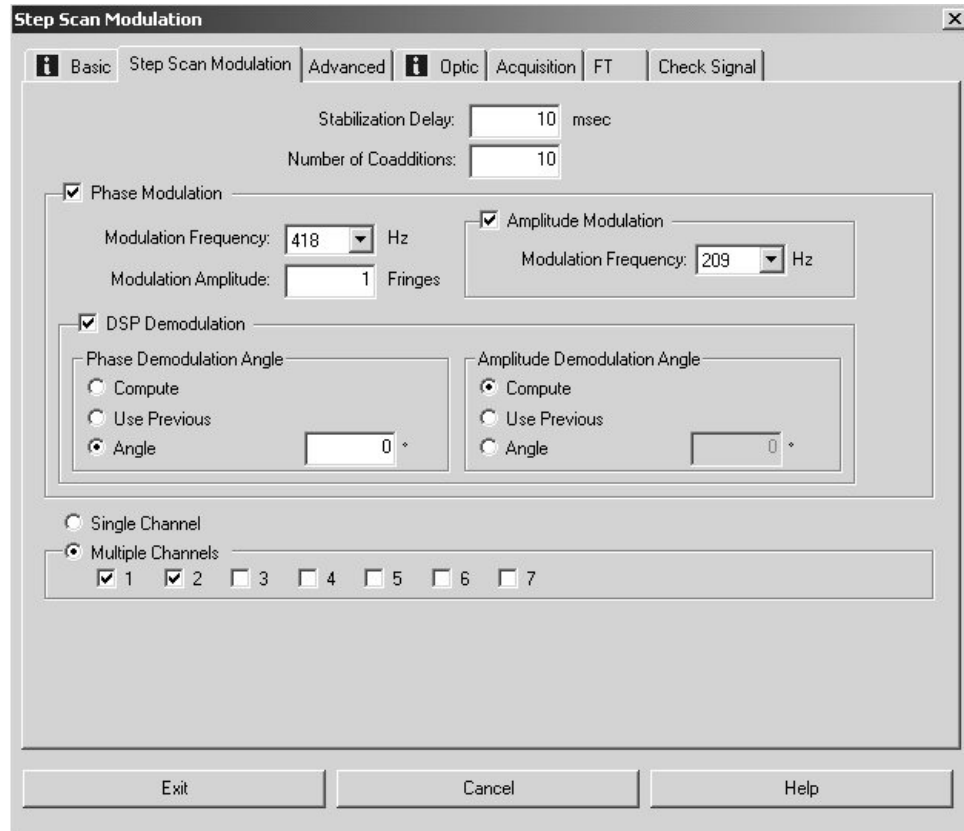
Figure 1: Step Scan Modulation Dialog Box – Setting up the Measurement Parameters

Stabilization Delay

In the Step Scan mode the interferometer mirror moves in a stepwise manner to each position. After the interferometer mirror has reached a position, it requires a settling time to stabilize at that position. Then, the system waits for a user-defined period, the *Stabilization Delay*, before it starts the data acquisition. The *Stabilization Delay* is an additional time (in milliseconds) for the mirror to further stabilize after a step.

Note: Using an external lock-in amplifier (LIA), the stabilization delay should be set to at least six times of the lock-in time constant. When working with AC-coupled detectors the stabilization delay should be larger than the time constant of the AC-coupling link (typically $\geq$ 100 milliseconds are sufficient.)

Number of Coadditions

This parameter has different meanings depending on whether you use an external lock-in amplifier (LIA) or an internal digital signal processor (DSP).

External LIA: After the stabilization delay has elapsed, the output signal of the lock-in amplifier is digitized at 10 microseconds intervals according to the value entered in the field *Number of Coadditions*. Afterward the values are summed and averaged. For example, 2000 coadditions correspond to a digitization time of 20 milliseconds.

> **Internal DSP:** After the stabilization delay has elapsed, measure-
> ments are performed periodically according to the lowest modulation
> frequency and the value entered in the field *Number of Coadditions*.
> If, for example, the phase modulation frequency is 414 Hz and the
> amplitude modulation frequency 17 Hz, the measurement intervals
> are N-multiples of the period length of the 17 Hz-signal (1/17 s),
> with *N* being the *Number of Coadditions*. The period of the lowest
> modulation frequency is used as a minimum value.

Phase Modulation

If you activate this check box the internal phase modulation is performed auto-
matically. The interferometer mirror oscillates about the step position with the
specified modulation frequency and the specified modulation amplitude.

Modulation Frequency

The selection of the modulation frequency depends on the experiment. PAS
measurements allow the analysis of various sample layers by variations in the
modulation frequency: high frequencies are used to analyze sample layers near
the surface while low frequencies are suitable for analyzing deeper sample
regions.

The drop-down list *Modulation Frequency* contains all possible modulation fre-
quencies as integer values. Note that only discrete modulation frequencies are
available. So, if you enter a value instead of selecting an option, the entered
value is corrected automatically by the TC-20 controller. The actual applied
modulation frequency is stored with the instrument parameters in the created
file. The generated modulation frequency is available as a TTL-signal at the
Ref-out-BNC-plug of the I/O cable.

Modulation Amplitude

Performing a phase modulation changes the shape of the resulting spectrum.
The shape depends on the amplitude of the applied phase modulation. In fact,
the normal single channel spectrum appears to be multiplied by a first order
Bessel function $J_1(2\pi\upsilon\varepsilon)$ of which the argument contains the product of the
modulation amplitude ε and the wave number υ. This function is zero if
$\upsilon\varepsilon = 0$. The first and largest maximum is reached at $\upsilon\varepsilon = 0,29$ and the first
zero crossing occurs at $\upsilon\varepsilon = 0,61$. At higher wave numbers, this function
oscillates with decreasing amplitude around zero.

To ensure that the entire spectrum of interest is above the first zero crossing of
J_1, entering the correct modulation amplitude is of crucial importance. Setting
the modulation amplitude to 1,0 fringe corresponds to a zero crossing of J_1 at
$9638cm^{-1}$, while a modulation amplitude value $\varepsilon = 2$ fringes causes a zero
crossing at $4819cm^{-1}$, etc. The highest signal response, however, is observed at
the largest possible amplitude. Therefore, the optimal modulation amplitude is a
compromise: choose the largest possible amplitude that will keep the spectral
region of interest above the first zero crossing of J_1.

Amplitude Modulation

In addition to the phase modulation (PM), a modulation signal for the amplitude modulation (AM) can be created that is accessed as a TTL voltage at the Aux-out-BNC-port of the I/O cable.

The amplitude modulation frequency is selected in the same way as the phase modulation frequency, as described above. The drop-down list *Modulation Frequency* contains all possible modulation frequencies as integer values. Note that only discrete modulation frequencies are available. So, if you enter a value instead of selecting an option, the entered value is corrected automatically by the TC-20 controller.

> **Note:** The higher frequency value f_{hi} of the two frequencies must always be an integral multiple $P = f_{hi}/f_{lo}$ of the lower frequency f_{lo}. Since both frequencies derive from the same quartz oscillator, the phase length of both signals does not change during the measurement.

DSP Demodulation

The option *DSP Demodulation* is only available if an internal phase modulation (PM) and amplitude modulation (AM) can be performed using an internal digital signal processor (DSP), this means that no lock-in amplifier (LIA) is required. This kind of demodulation allows the measurement signal demodulation of the PM-component as well as the AM-component.

For each measurement channel two single channel data blocks are generated as the result of a PM-demodulation. These data blocks are marked with *R* and *I*. *R* represents the component of the detector signal that is in phase with the modulation signal (in-phase), while *I* represents the component of the detector signal which is shifted 90 degrees from the modulation signal (quadrature). If both the the phase modulation and the amplitude modulation are performed during the measurement, the amplitude demodulation also generates a *R*- and *I*-data block representing the *in-phase* and *quadrature* component of the AM-signal.

Demodulating two frequencies, the system acts like two lock-in amplifiers connected in series: first, the component with the higher frequency f_{hi} is demodulated, then the component with the lower frequency f_{lo}.

Phase Demodulation Angle / Amplitude Demodulation Angle

The OPUS/STEP software package allows you to define the phase demodulation angle as well as the amplitude demodulation angle. To do this, you can choose between the following options:

Compute

If you select this option the program automatically calculates the phase angle φ between the modulation signal and the detector signal. Since the modulation signal is not digitized during the measurement, the phase angle cannot be computed by a direct comparison of detector signal and modulation signal. Instead, it is computed from the

assumption that the *in-phase* component is much larger than the *quadrature* contribution. Thus, the program finds a phase rotation by an angle φ that maximizes the *in-phase* component. This angle φ, maximizing the *in-phase* component, is the demodulation angle.

The in-phase component and the quadrature component, found in this way, can still be subject to further manual phase rotations by the angle δ. To do this, select in the OPUS *Manipulate* menu the function *Spectrum Calculator* and apply the following formulae:

$$R'(\varphi + \delta)= R(\varphi) \times \cos(\delta) - I(\varphi) \times \sin(\delta)$$

and

$$I'(\varphi + \delta)= R(\varphi) \times \sin(\delta) - I(\varphi) \times \cos(\delta)$$

Note: If you perform this transformation often we recommend working with a macro. The macro editor allows the use of *R* and *I* data blocks.

Use previous

This option allows you to use the stored phase angle of the previous measurement. Select this option when you measure a reference sample (e. g. carbon black) with a defined phase angle first, and then use the phase angle of this reference sample for the following measurements (e. g. phase-resolved DSP experiment). The phase angle remains stored in the acquisition processor until it is overwritten by a new one that is calculated with the *Compute* option.

Note: Since this program can only recognize one phase angle, this option cannot be used for measurements performed simultaneously with multiple channels because all channels use the same phase angle.

Angle

This option allows you to enter the desired phase angle manually (as a numerical value in degrees). Select this option if a phase angle has been determined from the instrument parameters of previous measurements.

Note: Since this program can only recognize one phase angle, this option cannot be used for measurements performed simultaneously with multiple channels because all channels use the same phase angle.

Single Channel / Multiple Channels

The OPUS/STEP software package allows you to choose between the options *Single Channel* and *Multiple Channels*.

If you perform measurements with a lock-in amplifier, the IR-beam is coupled to that detector which is defined in the standard measurement dialog box. Without changing the mirror position of the detector, up to seven electrical input channels for the detector multiplexer board can be selected, i.e. up to seven signals can be collected at the same time. This option is useful for collecting the in-phase and quadrature output signals of one or more external lock-in amplifiers. To do this, click on the *Multiple Channel* option button and check the desired input channels.

During the measurement the signals of these channels are collected subsequently. For each channel a separate result file is generated.

If you click on the *Single Channel* option button only one signal is collected from the standard input jack of the associated detector.

3.3 Starting the Measurement

To start the measurement click on the *Basic* tab and then on the *Start Step Scan Modulation Measurement* button.

If the parameters *Resolution, Phase Resolution*, *Wanted Low / High Frequency Limit* and *Acquisition Mode* have not been changed and the spectrometer is still in the step-scan mode when the measurement is started, the mirror is moved to the start position and the measurement starts. Otherwise, the spectrometer may switched to the rapid scan mode in order to set all relevant parameter and afterwards it is switched back to the step scan mode.

The status bar displays the actual operation mode of the spectrometer and the current mirror position. You can interrupt or terminate a measurement by right-clicking on the status bar and selecting either *Stop task* or *Abort task*. If a measurement is terminated prematurely the missing data points are added with the intensity value of the last data point. After the measurement the spectrometer remains in the step-scan mode.

The raw data are calculated using the FT-parameter defined in the standard *Measurement* dialog box and then stored automatically in the desired data block type.

> **Note:** If you have activated the *Use previous* option button(s), do not switch between the rapid scan mode and the step scan mode when performing the reference and the sample measurement. Otherwise, the absolute start position of the mirror can slightly be different.

3.4 Overflow Detection during the Measurement

If the signal exceeds the ADC input voltage range, the following error message appears:

<div align="center">Signal too large for ADC, overflow!</div>

This means that the measurement signal is larger than the dynamic range of the analog-to-digital converter (ADC). This can lead to large artifacts in the spectra. In this case abort the measurement. Before you repeat the measurement, reduce the amplification factor. To do this, click on the *Optic* tab and select a lower *Sample Signal Gain* value.

4 Time Resolved Step Scan with the internal ADC

4.1 General Information

This option allows the study of fast physical processes with a time resolution up to 5μsec using the standard internal analog-to-digital-converter (ADC). It requires special hardware and the OPUS/STEP software package. We also recommend the OPUS/3D software package to display the results in stacked-plot and contour-plot views.

During this fast type of time resolved measurement the interferogram is acquired in step scan mode by repeating the following procedure at every mirror position. As soon as the interferometer mirror has reached a new position, the mirror settles for a certain time specified by the parameter *Stabilization delay after stepping*. Then, *x* experiments are initiated and averaged (with *x* being the *Repetition/Coadd Count*). Between the experiments, the sample is allowed to recover for an *Experiments recovery time* (specified in milliseconds). During the experiment the sample is excited (e.g. flash of light or a quick field change) and the detector response to the perturbation is scanned in *N* timeslices (with *N* being the *Number of timeslices*), i.e. the changing ADC-signal is digitized *N* times at equidistant time intervals specified by the *Time resolution*. At the end of the measurement the data are sorted in *N* interferograms. Depending on the data blocks you have selected in the group field *Data blocks to be saved* on the *Advanced* page, either the interferograms or the spectra or both will be saved in the resulting 3D-file.

During the measurement the sample will be excited repeatedly *NPT* x *Repetition/Coadd Count* times (with *NPT* being the number of data points of the interferogram). Thus, it is necessary that the sample reacts reversibly to the excitation of the experiment and does not degrade.

4.2 Setting up the internal ADC

Before performing a time resolved measurement with the internal ADC you must set up this device. To do this, select in the OPUS *Measure* menu the *Optic Setup and Service* function. The *Optic Setup and Service* dialog box opens. Click on the *Devices/Options* tab. Activate the *Transient Recorder* check box (figure 2) and click on the *Setup* button.
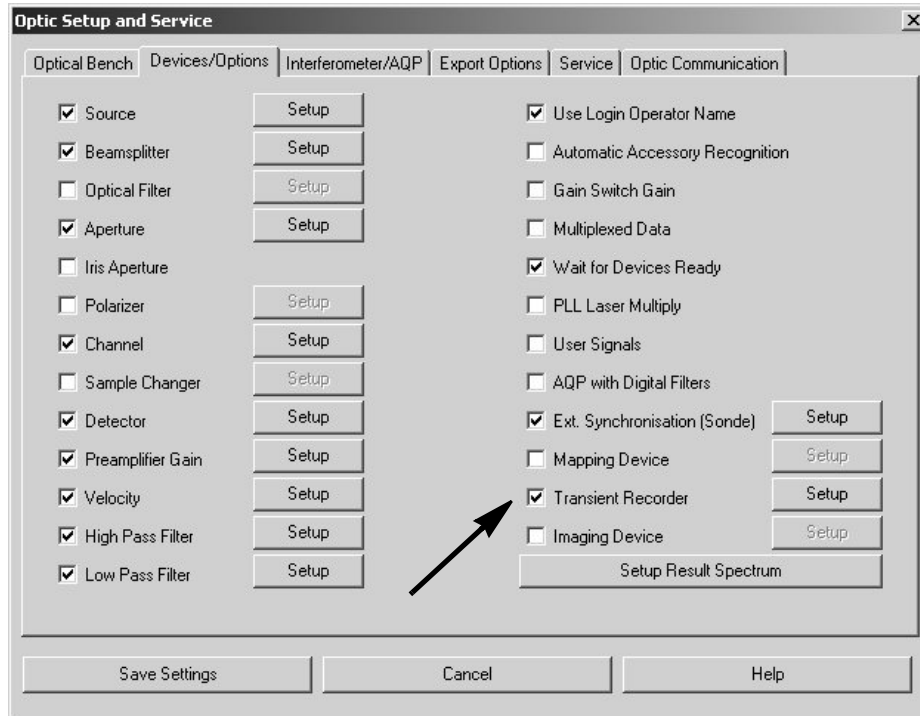
Figure 2: Optic Setup and Service Dialog Box - Setting up the internal ADC

The *Device/Options* dialog box (figure 3) opens. Make sure whether an entry containing the string *ADC* already exists in the list. If so, the internal ADC option is already set up. Otherwise, click on the *Add New Item* button and enter a string like *0=Internal ADC* (figure 3).

The number at the beginning of the string (*0* in the given example) must be entered; it indicates that the device in the list is unique. The string on the right hand side of the equals sign must contain the substring ADC; other additional substrings are optional. After entering the correct string, click on the *OK* button. On the *Optic Setup and Service* dialog (fig. 2) click on the *Save Settings* button.
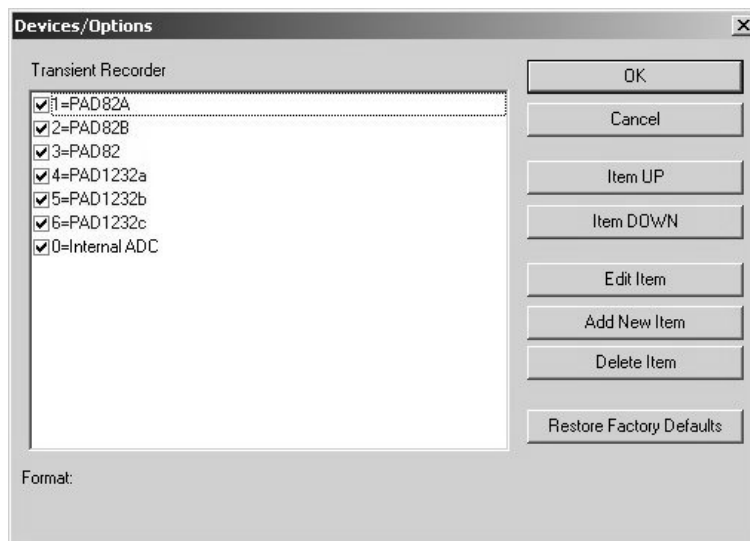

Figure 3: Devices/Options Dialog Box - Setting up the internal ADC

4.3 Setting up Measurement Parameters

Select in the OPUS *Measure* menu the *Time Resolved Step Scan* function. The *Step Scan Time Resolved Measurement* dialog box opens. Basically, this dialog box is identical to the *Measurement* dialog box (described in the OPUS Reference Manual), except for the *Recorder Setup* page.

Click on the *Recorder Setup* tab. The dialog window shown in figure 4 opens. It allows you to define the necessary parameters to perform a time resolved step scan measurement.

> **Note:** Before starting a measurement ensure that **all** parameters are set correctly. For detailed information on the measurement parameters of the other dialog windows refer to the OPUS Reference Manual.
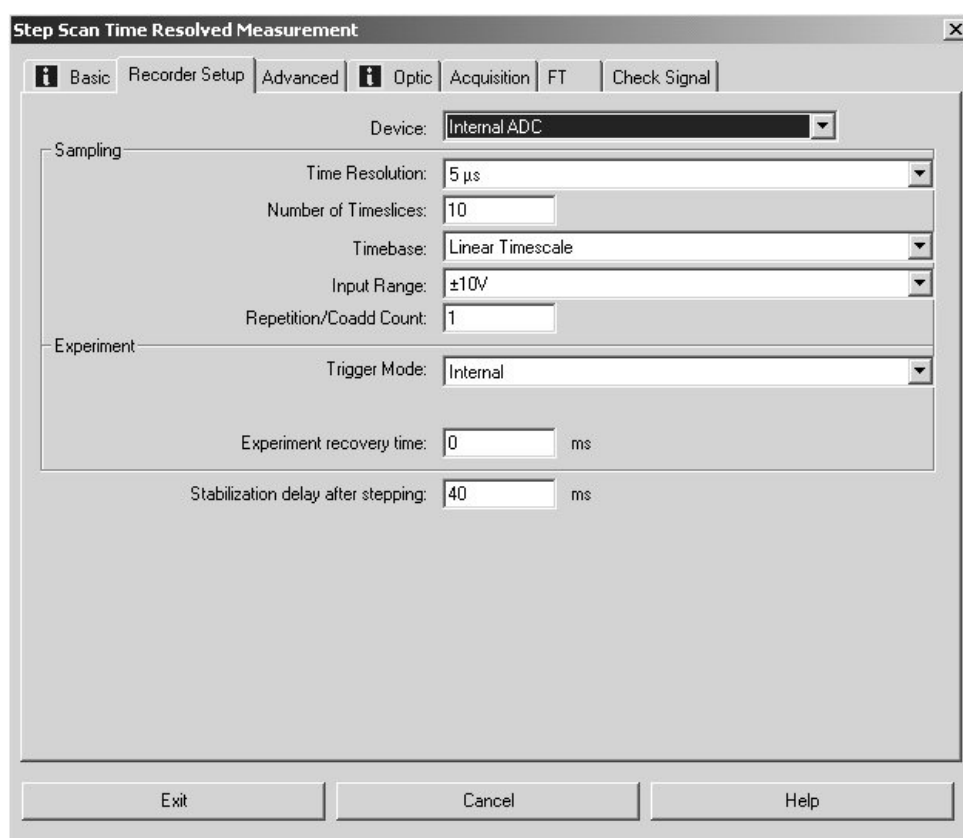

Figure 4: Step Scan Time Resolved Measurement Dialog Box - Recorder Setup

Device

Select the option *Internal ADC* from the *Device* drop-down list. Note that, once you have selected this option, the dialog window only displays those parameters being relevant for the internal ADC option.

> **Note:** This list contains all devices that are listed and checked in the *Devices/Option* dialog window (figure 3).

Time Resolution

In case of internal triggering the *Time Resolution* is the time interval between detector output digitizations and, consequently, also the time interval between subsequent timeslices or spectra. The maximum time resolution is 5μs. Do not enter a larger time resolution value, also in case of external triggering.

Number of Timeslices

This value represents the total number of interferograms measured with the specified time resolution. It also determines the total time the detector will detect the signal at a given interferometer mirror position. For example, if you set the time resolution to 10μs and define 20 timeslices, a total measurement time of 200μs is covered yielding 20 interferograms at 10μs intervals. For each timeslice a separate interferogram or spectrum is saved, depending on the data blocks you have selected in the *Data blocks to be saved* group field on the *Advanced* page.

Timebase

Possible settings are: *Linear Timescale* and *External*.

Linear Timescale

The linear timescale uses an internal clock that produces an equidistant time raster. Each resulting interferogram belongs to a time t that is multiple of the constant time resolution Δt, $t = n \times \Delta t$, with n being the running number of the interferogram.

External

This option allows you to apply an external signal which needs not be equidistant in time.

Input Range

In case of measurements with the internal ADC, the only possible setting is ±10 Volt. A signal having this voltage will fill the dynamic range of the ADC.

Repetition/Coadd Count

This value represents the number of data acquisitions to be performed at each mirror position. The purpose of coaddition is noise reduction. By increasing this value the signal-to-noise ratio can be improved to a certain degree. We recommend a value between 10 and 50. Note that a higher value (e.g. more than 100) does not improve the signal-to-noise ratio significantly because step scan measurements are sensitive to vibrations. So, a longer measurement time may have a negative effect on the spectrum due to external vibrations. Therefore, repeat the measurement several times instead of using a high coadd count value.

Trigger Mode

Possible settings are: *Internal*, *External Positive Edge* and *External Negative Edge*.

The experiment can be triggered either internally or externally. If you select the internal trigger mode, the excitation of the sample is started synchronously with the first digitization pulse. If you select an external trigger mode the digitization is performed after the specified edge of the experiment trigger is detected. Triggering can be set to occur either on the positive or negative going edge of the pulse.

Experiment Recovery Time

If the experiment is repeated several times, specify an *Experiment Recovery Time* to allow the sample, the source or the electronics to recover between the single measurements. The purpose of repeating the experiment is to improve the signal-to-noise ratio. The recovery time depends on the sample. It should be large enough to achieve identical repetitions.

Stabilization Delay after Stepping

The *Stabilization Delay after Stepping* is a wait time of the system allowing the mirror to stabilize after it has moved to a new position.

> **Note:** Do not confuse the *Stabilization Delay after Stepping* and the *Experiment Recovery Time*. The *Stabilization Delay after Stepping* runs only **one time** after each mirror step, while the *Experiment Recovery Time* runs **n times** (with *n* being the number of co-additions). Note that the *Stabilization Delay after Stepping* must be longer than the settling time of the detector and the amplifier. Using an AC-coupled amplifier set this value to at least 100ms.

4.4   Starting the Measurement

To start the measurement click on the *Basic* tab and then on the *Start Step Scan Time Resolved Measurement* button.

If the parameters *Resolution, Phase Resolution*, *Wanted Low / High Frequency Limit* and *Acquisition Mode* have not been changed and the spectrometer is still in the step-scan mode when the measurement is started, the mirror is moved to the start position and the measurement starts. Otherwise, the spectrometer may switched to the rapid scan mode in order to set all relevant parameter and afterwards it is switched back to the step scan mode.

The status bar displays the actual operation mode of the spectrometer and the current mirror position. You can interrupt or terminate a measurement by right-clicking on the status bar and selecting either *Stop task* or *Abort task*. If a measurement is terminated prematurely, the missing data points are added with the intensity value of the last data point. After the measurement the spectrometer remains in the step-scan mode.

The raw data are calculated using the defined FT-parameter and then stored automatically in the desired data block type containing the single time slices of the time resolved measurement in chronological order and the FT-parameters of measurement.

4.5   3D Correlation

To perform a 3D correlation the 3D software package is required. This software allows you to correlate 'dynamic spectra' that describe the spectral changes of a sample exposed to external perturbations that, in contrast to the modulation/demodulation technology, need not necessarily have the shape of a sine function. The result of a 3D correlation is a new 3D file. The correlation relation is illustrated in a 3D plot with two wave number axes. This type of plot allows you to determine the synchronous and asynchronous correlation spectra directly from the run time of the changes in the spectra without using a lock-in amplifier and a sine modulation frequency.

For detailed information refer to the 3D software manual.

5   Time Resolved Step Scan with a Transient Recorder

5.1   General Information

This option allows the study of extremely fast physical phenomena in the nano-second domain using a transient recorder. It requires special hardware and the OPUS/STEP software package. We also recommend the OPUS/3D software package to display the results in stacked-plot and contour-plot views.

During this fast type of time resolved measurement the interferogram is acquired in step scan mode by repeating the following procedure at every mirror position. As soon as the interferometer mirror has reached a new position, the mirror settles for a certain time specified by the parameter *Stabilization delay after stepping*. Then, x experiments are initiated and averaged (with x being the *Repetition/Coadd Count*). Between the experiments, the sample is allowed to recover for an *Experiments recovery time* (specified in milliseconds). During the experiment the sample is excited (e.g. flash of light or a quick field change) and the detector response to the perturbation is scanned in N time slices (with N being the *Number of timeslices*), i.e. the changing ADC-signal is digitized N

times at equidistant time intervals specified by the *Time resolution*. At the end of the measurement the data are sorted in *N* interferograms. Depending on the data blocks you have selected in the group field *Data blocks to be saved* on the *Advanced* page, either the interferograms or the spectra or both will be saved in the resulting 3D-file.

During the measurement the sample will be excited repeatedly *NPT* x *Repetition/Coadd Count* times (with *NPT* being the number of data points of the interferogram). Thus, it is necessary that the sample reacts reversibly to the excitation of the experiment and does not degrade.

5.2 Setting up the Transient Recorder

Before performing a time resolved measurement with a transient recorder you must set up the transient recorder. To do this, select in the OPUS *Measure* menu the *Optic Setup and Service* function. The *Optic Setup and Service* dialog box opens. Click on the *Devices/Options* tab. Activate the *Transient Recorder* check box (figure 5) and click on the *Setup* button.
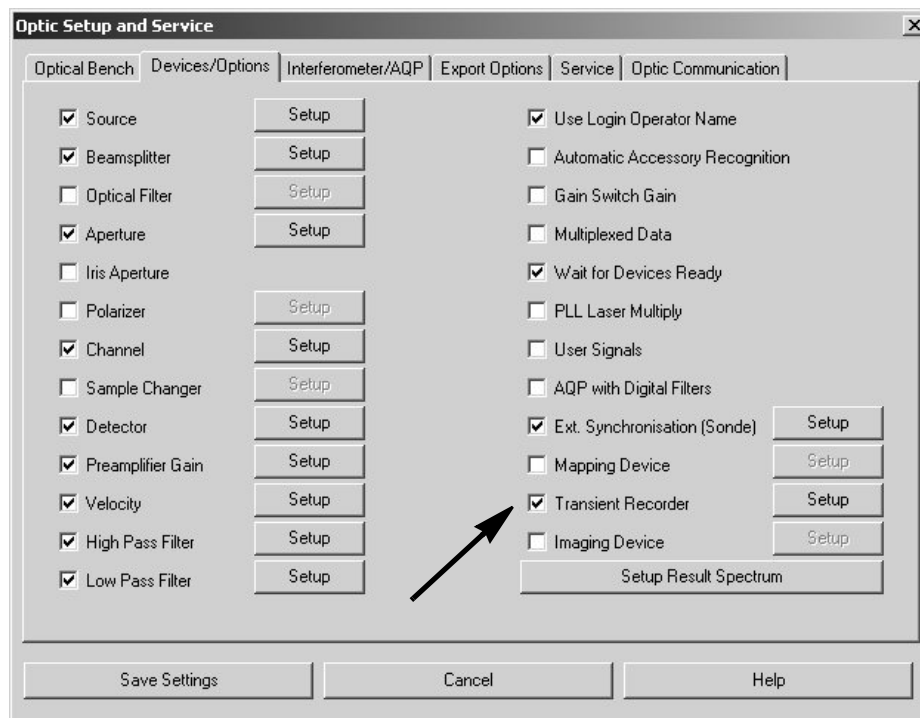

Figure 5: Optic Setup and Service Dialog Box - Setting up the Transient Recorder

The *Device/Options* dialog box (figure 6) opens. The available transient recorders that are displayed in the list depend on your spectrometer. Select the transient recorder that is connected to your spectrometer and uncheck all other options.

OPUS supports the following following transient recorders:

1) PAD82A
2) PAD82B
3) PAD82
4) PAD1232a
5) PAD1232b
6) PAD1232c

Note: The transient recorders (1) to (3) are equipped with the fast 8 bit-board and the transient recorders (4) to (6) have a dynamic range of 12 bits.

If no transient recorder is connected to your spectrometer, make sure that the *Transient Recorder* check box is not activated.
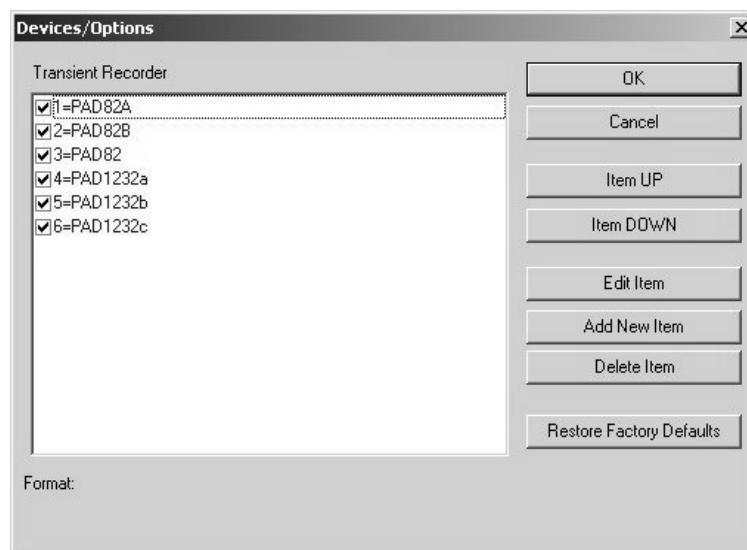

Figure 6: Devices/Options Dialog Box - Selecting the Transient Recorder

5.3   Setting up Measurement Parameters

Select the *Time Resolved Step Scan* function in the *Measure* menu. The *Step Scan Time Resolved Measurement* dialog box opens. Basically, this dialog box is identical to the *Measurement* dialog box (described in the OPUS Reference Manual), except for the *Recorder Setup* page.

Click on the *Recorder Setup* tab. The dialog window shown in figure 7 opens. It allows you to define the necessary parameters to perform a time resolved step scan measurement.

Note: Before starting a measurement ensure that **all** parameters are set correctly. For detailed information on the measurement parameters of the other dialog windows refer to the OPUS Reference Manual.
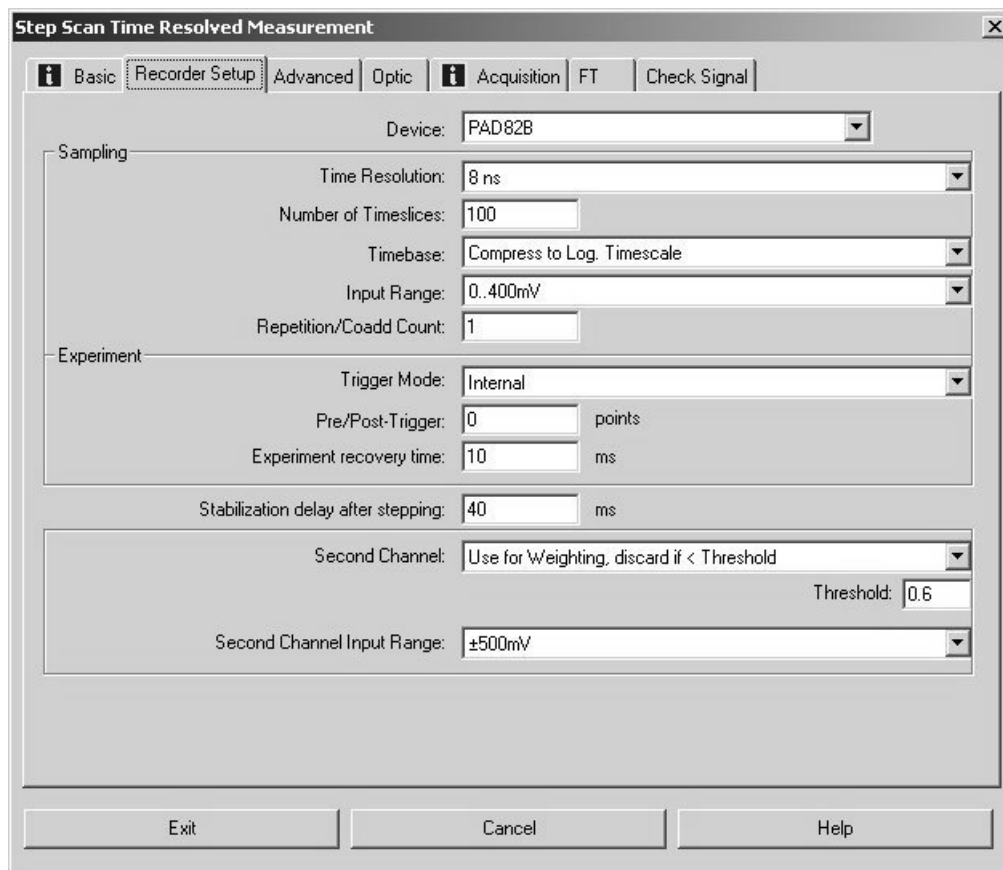
Figure 7: Step Scan Time Resolved Measurement Dialog Box - Recorder Setup

Device

Select the transient recorder that is actually connected to your spectrometer from the *Device* drop-down list. This list contains all devices that are listed and checked in the *Devices/Option* dialog window (figure 6).

Time Resolution

In case of internal triggering the *Time Resolution* is the time interval between detector output digitizations and, consequently, also the time interval between subsequent timeslices or spectra. The maximum time resolution is 5μs. Do not enter a larger time resolution value, also in case of external triggering.

Number of Timeslices

This value represents the total number of interferograms measured with the specified time resolution. It also determines the total time the detector will detect the signal at a given interferometer mirror position. For example, if you set the time resolution to 10μs and define 20 timeslices, a total measurement time of 200μs is covered yielding 20 interferograms at 10μs intervals. For each timeslice a separate interferogram or spectrum is saved, depending on the data blocks you have selected in the *Data blocks to be saved* group field on the *Advanced* page.

Timebase

Possible settings are: *External, Linear Timescale* and *Compress to Log. Timescale*.

External

This option allows you to apply an external signal which needs not be equidistant in time.

Linear Timescale

The linear timescale uses the internal clock of the transient recorder that produces an equidistant time raster. Each resulting interferogram belongs to a time *t* that is multiple of the constant time resolution Δt, $t = n \times \Delta t$, with *n* being the running number of the interferogram.

Compress to Log. Timescale

This option also uses the internal clock of the transient recorder to produce a set of equidistant sampling points linear in time. Then, the sampling points are averaged along the time axis so that an equidistant time axis results.

Input Range

Depending on the selected transient recorder the following options are possible: ± 200 mV, ± 500 mV, ± 1V, $0..400$mV, $0..2$V. Upon specifying the *Input Range*, bear into mind that the maximum signal fills the dynamic range of the ADC of the transient recorder in the best possible way.

Repetition/Coadd Count

This value represents the number of data acquisitions to be performed at each mirror position. The purpose of coaddition is noise reduction. By increasing this value the signal-to-noise ratio can be improved to a certain degree. We recommend a value between 10 and 50. A higher value (e.g. more than 100) does not improve the signal-to-noise ratio significantly because step scan measurements are sensitive to vibrations. So, a longer measurement time may have a negative effect on the spectrum due to external vibrations. Therefore, repeat the measurement several times instead of using a high coadd count value.

Trigger Mode

Possible settings are: *Internal, External Positive Edge* and *External Negative Edge*.

The experiment can be triggered either internally or externally. If you select the internal trigger mode, the excitation of the sample is started synchronously with the first digitization pulse. If you select an external trigger mode the digitization is performed after the specified edge of the experiment trigger is detected. Triggering can be set to occur either on the positive or negative going edge of the pulse.

Pre/Post-Trigger

The entry of a positive value (N>0) causes the initiation of the data acquisition after N timeslices have elapsed after triggering. The entry of a negative value (N<0) allows data acquisition of N timeslices before the trigger output (only in case of internal triggering).

Experiment Recovery Time

If the experiment is repeated several times, specify an *Experiment Recovery Time* to allow the sample, the source or the electronics to recover between the single measurements. The purpose of repeating the experiment is to improve the signal-to-noise ratio. The recovery time depends on the sample. It should be large enough to achieve identical repetitions.

Stabilization Delay after Stepping

This stabilization delay is the wait time after the mirror has moved to the next position and has stabilized.

> **Note:** Do not confuse the *Stabilization Delay after Stepping* with the *Experiment Recovery Time*. *Stabilization Delay after Stepping* means that the experiment is delayed after a mirror step while the *Experiment Recovery Time* occurs before an stabilization delay begins. The *Stabilization Delay after Stepping* must be longer than the settling time of the detector and the amplifier. Using an AC-coupled amplifier set this value to at least 100ms.

Second Channel

The *Second Channel* drop-down list contains the following options: *Unused, Use for Phase Correction, Use for Weighting, Use for Weighting, discard if < Threshold, Discard Experiment if < Threshold*.

Unused

If the transient recorder is operated at its highest possible time resolution, this option may be the only one accepted by the dialog. In case of the PAD82B transient recorder board, for example, you set the max. time resolution to 4ns and then select another option than *Unused* from the *Second Channel* drop-down list, the *Second Channel* and the *Time Resolution* field get a red background. If you move the cursor over these fields the following error message appears: *This time resolution is only supported in single channel mode.* The reason therefore is that this board achieves its maximum speed only in the interleaved mode utilizing both ADCs for the same channel.

Use for Phase Correction

Normally, phase correction algorithms function properly only if the resulting spectrum has exclusively positive intensities. If this is not the case, the DC signal from the detector preamplifier can be digitized in the second channel of the

PAD board. As the DC signal yields a positive spectrum, it can be used for calculating the correct phase which is then used to correct the phase of the signal from the first channel.

Use second channel for weighting

If the intensity of the excitation signal is not constant, the response of the sample to the excitation will also vary proportionally. Therefore, it becomes desirable to compensate the variations. This can be done by digitizing the excitation signal in the second channel of the transient recorder and computing a correction factor using this digitized signal (more precisely, using only those parts of the excitation signal which are larger than 80% of its average signal). Then, the correction factor is used to correct the signal of the first channel.

Use for weighting, discard if < Threshold

This option is identical to the option *Use second channel for weighting* with one exception: in addition to the *Use second channel for weighting* option, it is checked whether the excitation signal is smaller than the threshold. If this is the case, the data of the current experiment is discarded. This is signaled by a beep of 1000Hz. The threshold value range is between 0.0 (no threshold) and 1.0 (maximum threshold). The value 1.0 corresponds to 'full positive scale', i.e. if the *Second Channel Input Range* is set to ±500 mV, for example, it corresponds to +500mV which, in case of a 8 bit-boards, is in accordance with 128 ADC counts. Useful threshold values must be smaller than 1.0.

Discard Experiment if < Threshold

This option does not normalize the signal of the first channel, but discards the data of the current experiment if the signal of the second channel is smaller than the threshold and repeats the measurement. This option is useful for cases in which the intensity of the excitation signal is quite reproducible but may sometimes fail completely, e.g. if a flash of light does not ignite.

5.4   Starting the Measurement

To start the measurement click on the *Basic* tab and then on the *Start Step Scan Time Resolved Measurement* button.

If the parameters *Resolution, Phase Resolution*, *Wanted Low / High Frequency Limit* and *Acquisition Mode* have not been changed and the spectrometer is still in the step-scan mode when the measurement is started, the mirror is moved to the start position and the measurement starts. Otherwise, the spectrometer may switched to the rapid scan mode in order to set all relevant parameter and afterwards it is switched back to the step scan mode.

The status bar displays the actual operation mode of the spectrometer and the current mirror position. You can interrupt or terminate a measurement by right-clicking on the status bar and selecting either *Stop task* or *Abort task*. If a measurement is terminated prematurely, the missing data points are added with the intensity value of the last data point. After the measurement the spectrometer remains in the step-scan mode.

The raw data are calculated using the defined FT-parameter and then stored automatically in the desired data block type containing the single time slices of the time resolved measurement in chronological order and the FT-parameters of measurement.

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