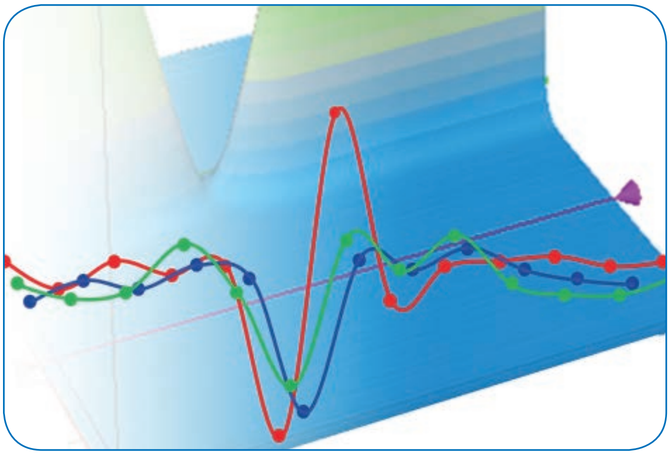

Application Note # AN132

Interleaved Time-Resolved FT-IR Spectroscopy

Time-resolved FT-IR spectroscopy (TRS) is widely used e.g. to track the kinetics of single shot chemical reactions via Rapid-Scan [1-4] or repeatable kinetics via the Step-Scan technique [5-9]. Although both techniques provide outstanding application opportunities they also imply inherent limitations.

Introduction

Depending on spectral resolution the Rapid-Scan technique is limited to a time resolution in the low millisecond range, not always sufficient for typical applications of interest. For repetitive kinetics Step-Scan overcomes these limitations and reaches a time resolution down to the low nanosecond range mainly limited by the rise time of the applied detector. On the other hand step-scan makes less efficient use of the available measurement time since the necessary mirror repositioning requires interrupting data acquisition and increases the fraction of dead time. Depending on the mode of amplification (AC or DC coupling), trigger scheme and time scale of the observed kinetics, the interferometer stepping rate may range from ca. 50 Hz down to a fraction of 1Hz.

Keywords	Instruments and software
FT-IR, FTIR infrared	Vertex series, VERTEX80, VERTEX80v
temporal resolved, spectroscopy	OPUS spectroscopic software
Step-Scan, S510	OPUS/3D
Rapid-Scan, S129	
Interleaved	Interleaved script S511

Interleaved Scan Principle

Interleaved time-resolved FT-IR spectroscopy is a technique for a certain class of repetitive kinetics, using the measurement time much more efficiently than Step-Scan. The interferometer mirror is moving continuously and zero-crossings of the internal HeNe laser are used to trigger the experiment. Data are not only acquired at the HeNe zero crossings (corresponding to t=0) but also at mirror positions in between, belonging to certain time delays Δt. Hence the system continuously acquires data and in principle a full series of time-resolved interferograms can be collected within one single scan. The temporal sequence of interleaved data acquisition is shown in fig. 1, explaining how additional interferograms (green and blue curve) are created in between the common data acquisition triggers (vertical red lines).

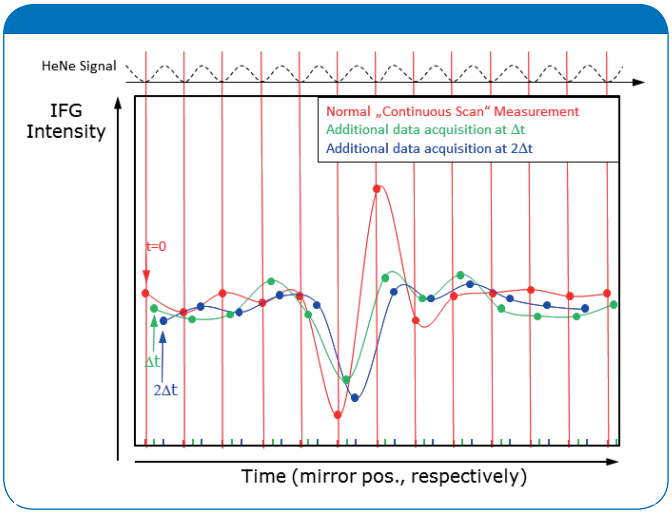

Figure 1: Regular data acquisition start triggers derived from the HeNe laser control signal shown on top. Red curve: "Normal" continuous scan interferogram representing the kinetics start time t=0. Green curve and blue curve: interleaved acquired interferograms at time delay Δt (green) and 2x Δt (blue).

Although the interleaved approach is not completely new [10] and was available for the famous Bruker IFS66v/S and IFS66/S spectrometers already it did so far not establish itself as widely accepted technique. The main reason are the technical limitations of last millennium's FT-IR spectrometers: in those days electronics as well as the required stability of scanning speed and software interfaces may not have completely met the requirements of high performance interleaved FT-IR spectroscopy. In fact with modern high-end research FT-IR spectrometers such as e.g. the VERTEX80(v), these limitations have been overcome, making interleaved spectroscopy a powerful alternative.

Measurement Example

Here we demonstrate the "renewed" interleaved TRS technology using the VERTEX 80 and its vacuum version VERTEX 80v with state of the art electronics and interferometer technology. By means of time resolved measurement of the emission of a pulsed infrared LED the outstanding performance of the interleaved TRS technique is nicely confirmed. For the discussed experiment the signal-to-noise ratio (SNR) for interleaved acquisition (see fig. 2 and 3) is significantly higher compared to Step-Scan and almost similar to continuous scan acquisition in case of constant LED emission (fig. 3).

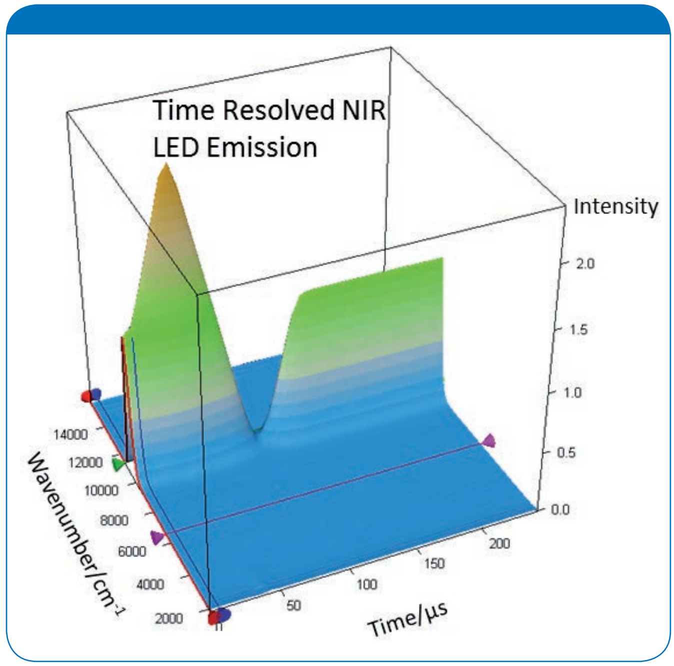

Figure 2: Time-resolved emission of an LED with time profile controlled by a function generator, measured via interleaved FT-IR spectroscopy. Also in case of averaging many interleaved scans the time profile is preserved and does not smear out, proving the accuracy of interleaved data acquisition.

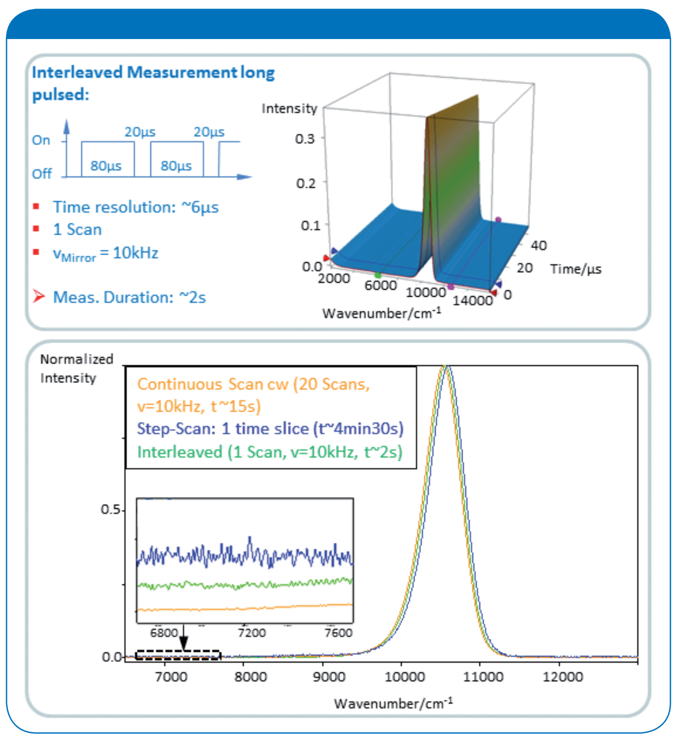

Figure 3: Interleaved time resolved measurement of the same NIR LED, but with rectangular time profile. Lower side inset: comparison of achieved noise level with interleaved acquisition compared to non-averaged Step-Scan and non-kinetics continuous scan spectrum. Note the different experimental measurement times of ~ 4 1/2 min for step-scan and 2s only for the interleaved scan TRS experiment.

As shown in figure 4 with increasing number of interleaved scans the SNR can be increased further. The dedicated script S511, available from Bruker as well, allows to comfortably average an arbitrary number of interleaved scans. This again indicates the high potential of interleaved TRS thanks to the extreme stability of the VERTEX data acquisition technique and DigiTect detector technology, described in the related patents [11].

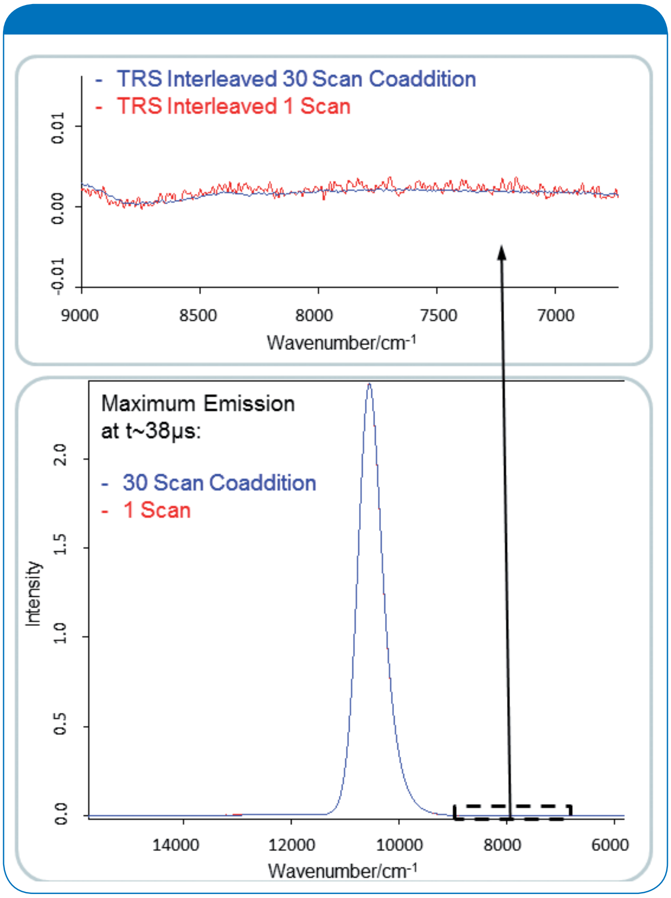

Figure 4: NIR LED interleaved time resolved measurement showing the achieved noise level in case of averaging 30 scans. The strong improvement compared to 1 scan implies the absence of relevant systematic noise and proves the high data acquisition stability of the VERTEX 80 series.

Conclusion

The presented measurement data proves the high performance of interleaved time resolved FT-IR spectroscopy with VERTEX series spectrometers. Nevertheless this new generation of interleaved spectroscopy shall not be understood as general replacement for the step-scan technique. Regarding flexibility due to its huge parameter space and various powerful modifications such as AC coupled differential spectroscopy or modulation techniques, step-scan remains definitely state of the art. However for a certain class of repeatable experiments interleaved FT-IR spectroscopy can be a powerful and easy to use alternative with clear advantages regarding signal to noise ratio and/or measurement duration. Finally it is worth to mention that interleaved scan TRS is unique for the VERTEX FT-IR spectrometer series and no other FT-IR manufacturer on the market is currently able to offer it. Again this is an example for the technology leadership of Bruker in the field of high-end research FT-IR.

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