

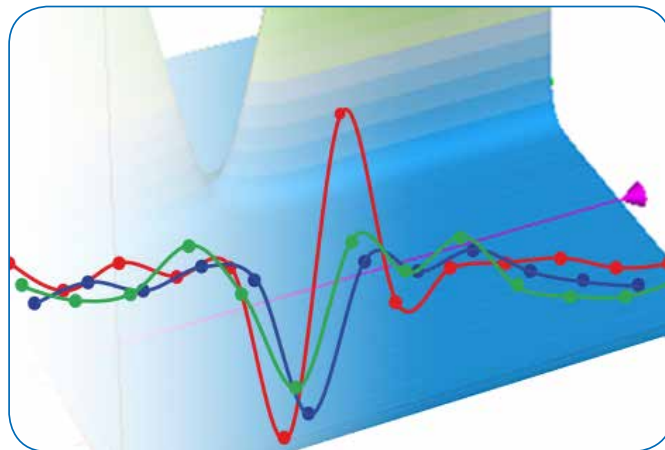
Application Note AN M132

Interleaved Time-Resolved FTIR Spectroscopy

Time-resolved FTIR spectroscopy (TRS) is widely used to characterize 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 have 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 many applications of interest. For repetitive kinetics, step-scan overcomes these limitations and reaches a time resolution down to the low nanosecond range and is 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 the data acquisition and increases the amount 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. 50Hz down to a fraction of 1 Hz.



Keywords

FTIR, FTIR infrared

temporal resolved, spectroscopy

Step-Scan, S510

Rapid-Scan, S129

Instruments and software

INVENIO and VERTEX series, VERTEX 80, VERTEX 80v

OPUS spectroscopic software

OPUS/3D

Interleaved Scan Principle

Interleaved time-resolved FTIR spectroscopy is a technique applicable to a certain class of repetitive kinetics, where the measurement time is used much more efficiently compared to step-scan. The interferometer mirror is moving continuously and zero-crossings of the internal HeNe laser are used to trigger the experiment. Data is 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 curves) are created in between the common data acquisition triggers (vertical red lines).

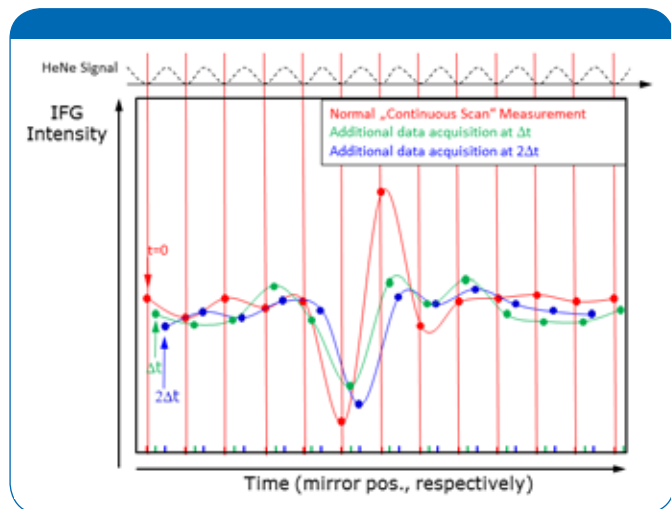


Figure 1: Regular data acquisition start triggers derived from the HeNe laser control signal (overlaid vertical lines). Red curve: “Normal” continuous scan interferogram representing the kinetics start time $t=0$. Green curve and blue curves: interleaved acquired interferograms at time delays Δt (green) and $2x \Delta t$ (blue).

Although the interleaved approach is not completely new [10] and was previously available for the popular Bruker IFS66v/S, IFS66/S, and IFS120HR spectrometers, it was not a widely utilized technique. The main reason was the technical limitations of the last millennium’s FTIR spectrometers. The electronics, required stability of the scanning mirror, and software interface may not have completely met the requirements of high performance interleaved FTIR spectroscopy. In fact with modern high-end research FTIR spectrometers, such as the VERTEX 80v, these limitations have been overcome, making interleaved spectroscopy a powerful option for investigating kinetic processes

Measurement Example

Here we demonstrate the “renewed” interleaved TRS technique using the VERTEX 80 and its vacuum version the 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 demonstrated. For this experiment the signal-to-noise ratio (SNR) for interleaved acquisition (see fig. 2 and 3) is significantly higher than that achievable by step-scan and similar to that obtained by continuous scan acquisition.

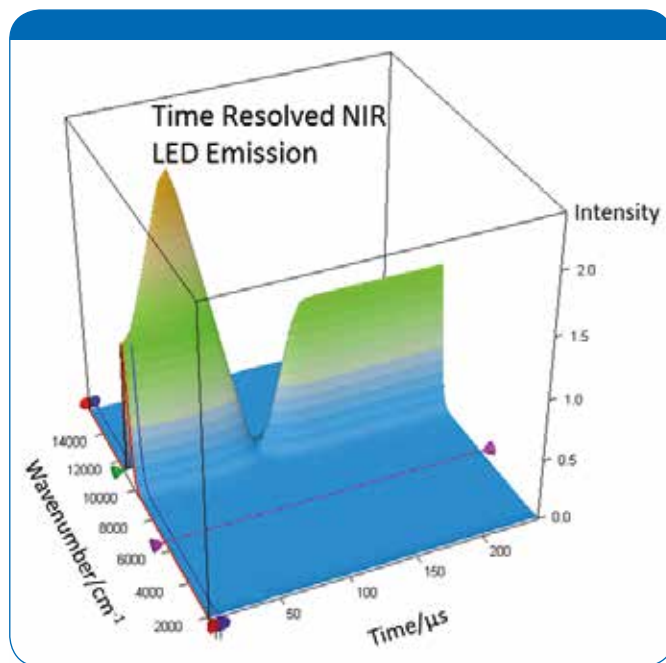


Figure 2: Time-resolved emission of an LED with time profile controlled by a function generator and measured via interleaved FTIR spectroscopy. Note that when averaging many interleaved scans, the time profile is preserved, proving the accuracy and reproducibility of interleaved data collection.

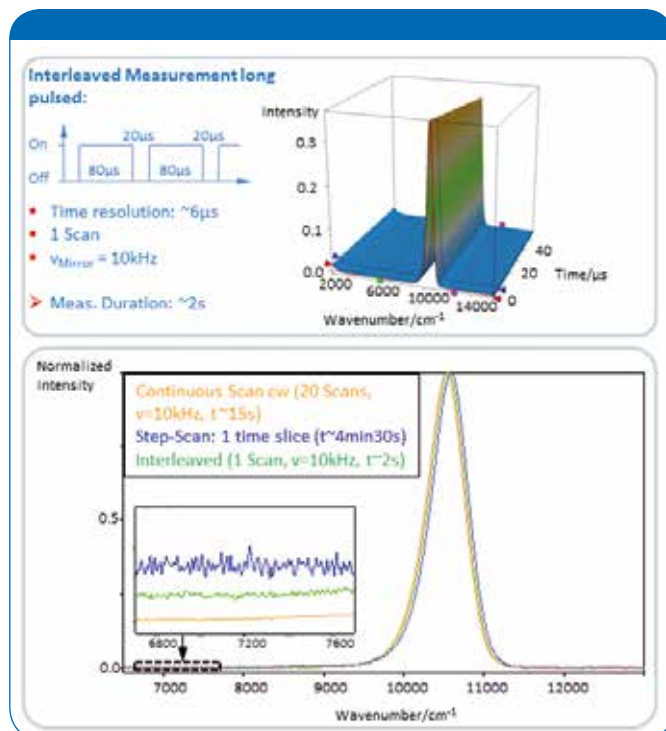


Figure 3: Interleaved time resolved measurement of the same NIR LED, but with a rectangular time profile. Lower left inset: comparison of achieved noise level with interleaved acquisition (middle), non-averaged step-scan (top), and continuous scan (bottom). Note the different experimental measurement times of ~ 4 1/2 min for step-scan and 2s only for the interleaved scan TRS experiment.

As illustrated in fig. 4, increasing the number of interleaved scans yields the expected improvement in SNR. The dedicated script S511, available from Bruker as well, allows the user to set the number of interleaved scans. This again indicates the potential of interleaved TRS, due to the extreme stability of the data acquisition and DigiText detector technology of INVENIO R and VERTEX series spectrometers as described in the related patents [11].

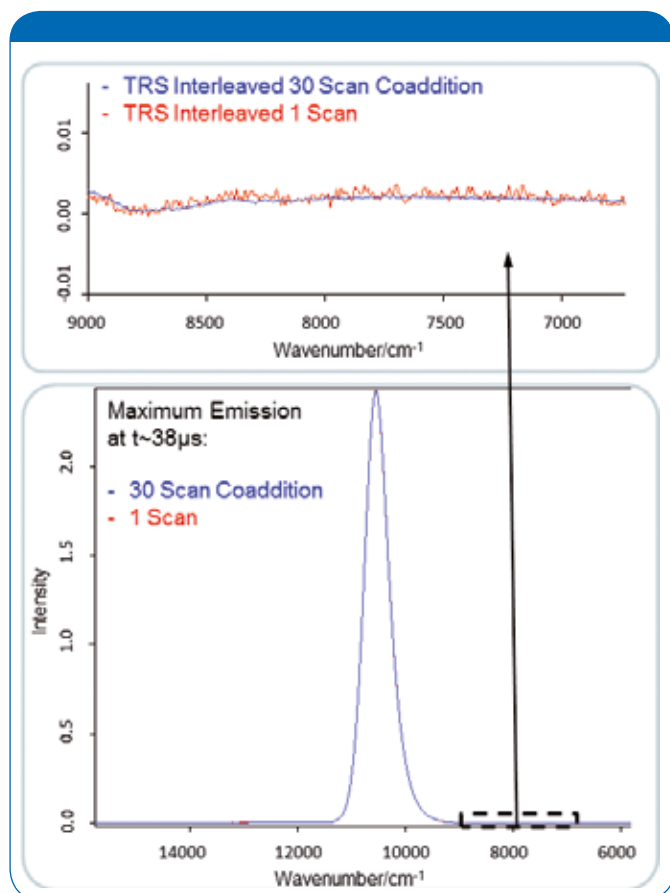


Figure 4: NIR LED interleaved time resolved measurement showing the achieved noise level in case of averaging 30 scans. The strong improvement compared to 1 scan implies the absence of relevant systematic noise and proves the high data acquisition stability of the VERTEX 80 series.

Conclusion

The presented measurement data demonstrates the high performance of interleaved time resolved FTIR spectroscopy with the INVENIO R and VERTEX series spectrometers. This new generation of interleaved spectroscopy is complimentary to the step-scan technique and is most applicable where many repeated measurements are not possible. Step-scan TRS is still capable of data collection orders of magnitude faster. Step-scan can also be utilized for other state-of-the-art experiments such as AC coupled differential spectroscopy as well as amplitude and phase modulation based data collections. However for a certain class of repeatable experiments, interleaved FTIR 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 mentioning that interleaved scan TRS is unique for the INVENIO R and VERTEX FTIR spectrometer series and no other FTIR manufacturer on the market offers this capability.

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● Bruker Optik GmbH

Ettlingen · Deutschland
Phone +49 (7243) 504-2000
Fax +49 (7243) 504-2050
info.bopt.de@bruker.com

Bruker Optics Inc.

Billerica, MA · USA
Phone +1 (978) 439-9899
Fax +1 (978) 663-9177
info.bopt.us@bruker.com

Bruker Shanghai Ltd.

Shanghai · China
Phone +86 21 51720-890
Fax +86 21 51720-899
info.bopt.cn@bruker.com

www.bruker.com/optics

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