

Time-of-flight spectroscopy up to 1400 nm for analysis of turbid media

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Abstract: A system capable of performing near infrared time-of-flight spectroscopy (TOFS) up to 1400 nm for analysis of turbid materials is described, and first results are reported.

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1. Introduction

Due to the relatively weak absorption of overtone and combinational bands found in the near infrared spectral region, spectroscopy in this range offers deep light penetration and thus the rather unique possibility of performing non-destructive and non-invasive analysis of solid and liquid samples (no, or limited, need for sample preparation). This advantage has made near infrared spectroscopy (NIRS) a very important technique for analysis of important materials such as foods and agricultural products [1], biological tissue [2, 3], and pharmaceutical solids [4]. A general complication is, however, that many of these materials are highly scattering (turbid). This means that optical pathlengths are unknown, and that detected intensities varies strongly with physical properties (not only chemical content). As a results, obtained spectra cannot be analysed using standard Beer-Lambert-Bouguer analysis. Instead, NIRS often rely on sophisticated calibration schemes, in which all possible variations in samples structure (scattering) and chemical content are to be covered. In contrast, photon time-of-flight spectroscopy (PTOFS) is a tool for analysis of highly scattering (turbid) materials that can distinguish between the effects of absorption and scattering, thus allow calibration free quantitative assessment of chemical content. The technique has proven very valuable in medical applications (tissue optics) [3], and is a promising tool also for applications in pharmaceutical analysis [5].

A severe limitation of PTOFS has been the limited spectral range covered. While earlier work has not reached above 1100 nm [6, 7, 8], we have now extended the spectral range covered by PTOFS up to 1400 nm. The extension allows interaction with the rich flora of important and diagnostic overtone and combinational bands found above 1100 nm, thus significantly increasing the applicability of PTOFS for analytical chemistry of turbid materials.

2. Instrumentation

Our new PTOFS spectrometers is built around a supercontinuum (SC) fiber laser (Fianium SC 500-6) and fast micro-channel plate photomultiplier tubes (MCP-PMTs). The SC source supplies pulses of light in the 500 to 2000 nm region with a duration of less than 50 ps FWHM. Acousto optical tunable filters (AOTFs, AA Crystal Technology) are used to isolate narrow bandwidth pulses (typically below 8 nm FWHM). In order to perform time-resolved detection in the 1100-1400 nm range, we employ a sophisticated InP/InGaAsP MCP-PMT (R3809-68, Hamamatsu Photonics). Detection below 1100 nm is handles by a second MCP-PMT (R3809-59, Hamamatsu Photonics). Photon time-of-flight distributions are obtained by means of time-correlated single photon counting (TCSPC; SPC-300 Becker&Hickl). The systems is shown in Figure 2.

3. Results

The capability of our instrument is here demonstrated by investigating the optical properties of an TiO₂-baed epoxy phantom designed to mimic the extreme level of scattering exhibited by pharmaceutical tablets [9]. The sample is of cylindrical shape (10 mm in diameter, and 3 mm thick). Optical properties are derived by employing the theory of light diffusion in slabs [10]. The system IRF are measured and taken into account. A summary of optical properties are shown in Figure 2. Further data will presented at the conference.

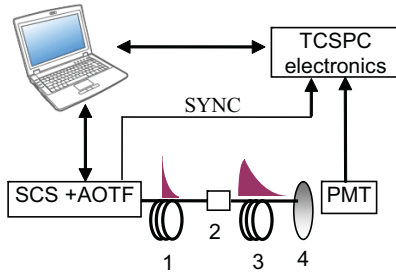


Fig. 1. Schematic of the spectrometer: 1) grad. index excitation fiber, 2) sample, 3) gradient index collection fiber, 4) ND filter wheel

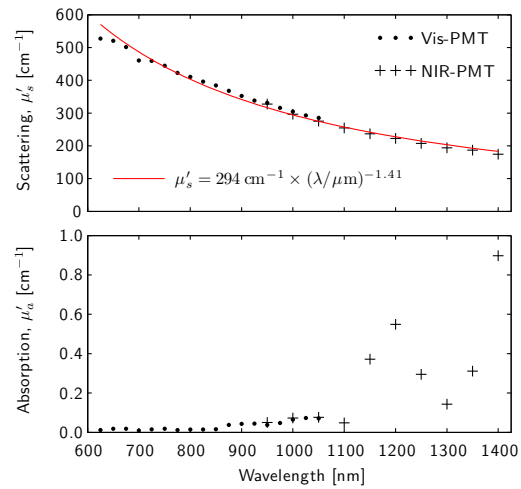


Fig. 2. Summary of optical properties. The smooth decay with wavelength of the reduced scattering coefficient indicate good system performance. The prominent and absorption features above 1100 nm are mainly related to overtone CH and OH bands, and illustrates the value of moving to longer wavelengths.

4. Discussion

The extension of the spectral range covered by PTOFS to cover the range from 1100 to 1400 nm is an important step in order to increase the applicability of the technique. Promising areas of applications include for example tissue diagnostics, clinical chemistry and analysis of intact pharmaceutical solids (the latter today often done by steady-state NIRS in the region now reached by PTOFS). Note that the specific use of the newly accessed region for tissue applications remains to be explored, but may include for example lipid spectroscopy or glucose monitoring.

References

1. Haibo Huang, Haiyan Yu, Huirong Xu, and Yibin Ying, "Near infrared spectroscopy for on/in-line monitoring of quality in foods and beverages: A review," *J. Food Eng.* **87**, 303-313 (2008).
2. G.A. Millikan, "The oximeter, an instrument for measuring continuously the oxygen saturation of arterial blood in man," *Rev. Sci. Instr.* **13**, 434-444 (1942).
3. A.J. Welch and M.J.C. van Gemert, editors, *Optical-thermal response of laser-irradiated tissue*, (Plenum Press 1995).
4. M. Blanco, J. Coello, H. Iturriaga, S. MasPOCH, and C. de la Pezuela, "Near-infrared spectroscopy in the pharmaceutical industry," *Analyst* **123**, 135-150 (1998).
5. J. Johansson, S. Folestad, M. Josefson, A. Sparen, C. Abrahamsson, S. Andersson-Engels, and S. Svanberg, "Time-resolved nir/vis spectroscopy for analysis of solids: Pharmaceutical tablets," *Appl. Spectrosc.* **56**, 725-731 (2002).
6. C. Abrahamsson, T. Svensson, S. Svanberg, S. Andersson-Engels, J. Johansson, and S. Folestad, "Time and wavelength resolved spectroscopy of turbid media using light continuum generated in a crystal fiber," *Opt. Express* **12**, 4103-4112 (2004).
7. A. Bassi, J. Swartling, C. D'Andrea, A. Pifferi, A. Torricelli, and R. Cubeddu, "Time-resolved spectrophotometer for turbid media based on supercontinuum generation in a photonic crystal fiber," *Opt. Lett.* **29**, 2405-2407 (2004).
8. A. Pifferi, A. Torricelli, P. Taroni, D. Comelli, A. Bassi, and R. Cubeddu, "Fully automated time domain spectrometer for the absorption and scattering characterization of diffusive media," *Rev. Sci. Instr.*, **78**, 053103 (2007).
9. T. Svensson, M. Andersson, L. Rippe, S. Svanberg, S. Andersson-Engels, J. Johansson, and S. Folestad, "VCSEL-based oxygen spectroscopy for structural analysis of pharmaceutical solids," *Appl. Phys. B*, **90**, 345-354 (2008).
10. D. Contini, F. Martelli, and G. Zaccanti, "Photon migration through a turbid slab described by a model based on diffusion approximation: I. theory," *Appl. Opt.* **36**, 4587-4599 (1997).