

## Flow through FTIR sensor based on solid phase spectroscopy (SPS) on conventional octadecyl (C18) silica

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### ABSTRACT

The concept of a novel FTIR flow through sensor based on rigid silica C18 particles placed in a mid-IR flow cell is presented applied to the example of caffeine determination in soft drinks. The system is based on a standard flow cell equipped with two polyethylene spacers of different size that yield a gap inside the assembled flow cell. Particles of appropriate size ( $<25\ \mu\text{m}$  diameter) suspended in methanol are pumped into the assembled flow cell where the particles are retained at the gap formed by the two spacers. Using an automated sequential injection system, pre-conditioning, sample–sensor interaction and sorbent regeneration can be performed in a highly reproducible way. The characterization and validation results clearly demonstrate the capability of the developed flow through FTIR sensor to determine non-polar molecules, such as caffeine, at the parts per million level, with a linear range from 1.8 to 115  $\text{mg L}^{-1}$ , a precision expressed as R.S.D. of 4.1% and a sample throughput of approximately 10 samples/h. Furthermore, the sensor has the potential to be easily adapted to the analysis of polar, non-polar, cationic and anionic molecules by changing the solid support, providing interesting possibilities for the development of new applications in the trace analysis of organic molecules.

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### 1. Introduction

In 1976 Yoshimura et al. [1] proposed combining of an active solid support to pre-concentrate the analyte with direct measurement of the species of interest, retained onto the solid phase, by absorption spectroscopy. This methodology was firstly called ion-exchanger colorimetry which, from a more generic point of view, also has been called solid phase spectroscopy or solid phase spectrophotometry (SPS) [2]. The attention paid to SPS increased significantly when the solid phase retention unit was placed inside a flow cell allowing real time monitoring of the retention of the target analytes [3] originating flow injection-solid phase spectroscopy (FI-SPS). Therefore, the generic advantages of SPS, mainly sensitivity and selectivity, were combined with those related to flow methods: rapidity, automation and reduction of the reagent consumption [4]. The sensitivity and selectivity are due to the separation of the analyte from the matrix and its pre-concentration on the solid support, which is placed directly in the measurement cell of a non-destructive molecular spectroscopic detector.

FI-SPS systems have been widely employed with UV–vis detectors for the determination of inorganic [5–7] and organic

[8–10] analytes, by using different types of solid supports such as anionic and cationic resins, polar and non-polar solid supports. Furthermore, FI-SPS has also been achieved with near infrared (NIR) [11] and Raman spectroscopic detection [12,13].

Schindler pioneered the development of FI-SPS systems in the mid-IR spectral region demonstrating an increase in sensitivity by a factor of 40, on the example of acetate analysis, when compared to direct measurement of the aqueous acetate standards. In order to meet the short optical path lengths, dictated by the strong mid-IR absorption of aqueous sample, flexible agarose beads had to be used as the solid phase material placed in the flow cell. Using such flexible material it was possible to immobilize the beads in the flow cell by squeezing them between two windows approximately 50  $\mu\text{m}$  apart. Due to flexible nature of the beads tightness of the flow cell could also be assured. After the first work on malic and acetic acid [14] this concept was also successfully applied to other applications; such as the determination of wine astringency by retaining wine phenols on proteins immobilized in the flow cell [15], carbohydrates in beer employing the beads carrying immobilized maltase [16] and water hardness based on an indirect method [17] again using agarose or sephadex beads.

An alternative approach to construct a mid-IR based sensor system uses the attenuated total reflection (ATR) technique [18]. Here an appropriate chemical modification of the employed internal reflection elements (IREs) opens up the possibility of

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developing a wide range of different sensor approaches. Examples are polymer coated IRE elements for the enrichment and in situ measurement of chlorinated hydrocarbons [19] or pesticides, as well as IRE elements on which membranes are chemically bound in order to monitor molecular interactions or structural changes of the membrane upon interaction with the analyte [20].

Recently, a flow sensor based on a horizontal diamond IRE element, and using conventional, rigid, sorbent materials (LiChrolut-EN, a polystyrene–divinylbenzene-based material) has been introduced. Intense contact between the IRE element and the beads was achieved by pressing them against the IRE element and using a home-built flow through poly(tetrafluoroethylene) (PTFE) accessory for connection to the flow system. This sensor concept was used for the determination of caffeine [21] yielding a precision, established as relative standard deviation (R.S.D.), of 4%, a limit of detection (LOD) of  $7 \text{ mg L}^{-1}$  and a sample throughput of 4.3 samples/h. Here, we present a strategy for construction of flow through FTIR sensors also based on rigid sorbent materials (C18 silica beads) for transmission measurement for improved sensitivity and sample throughput. In this study, caffeine has again been selected as test molecule and

has been validated in terms of accuracy, precision, linearity and LOD. Furthermore, its utility and potential in real sample analysis has been demonstrated.

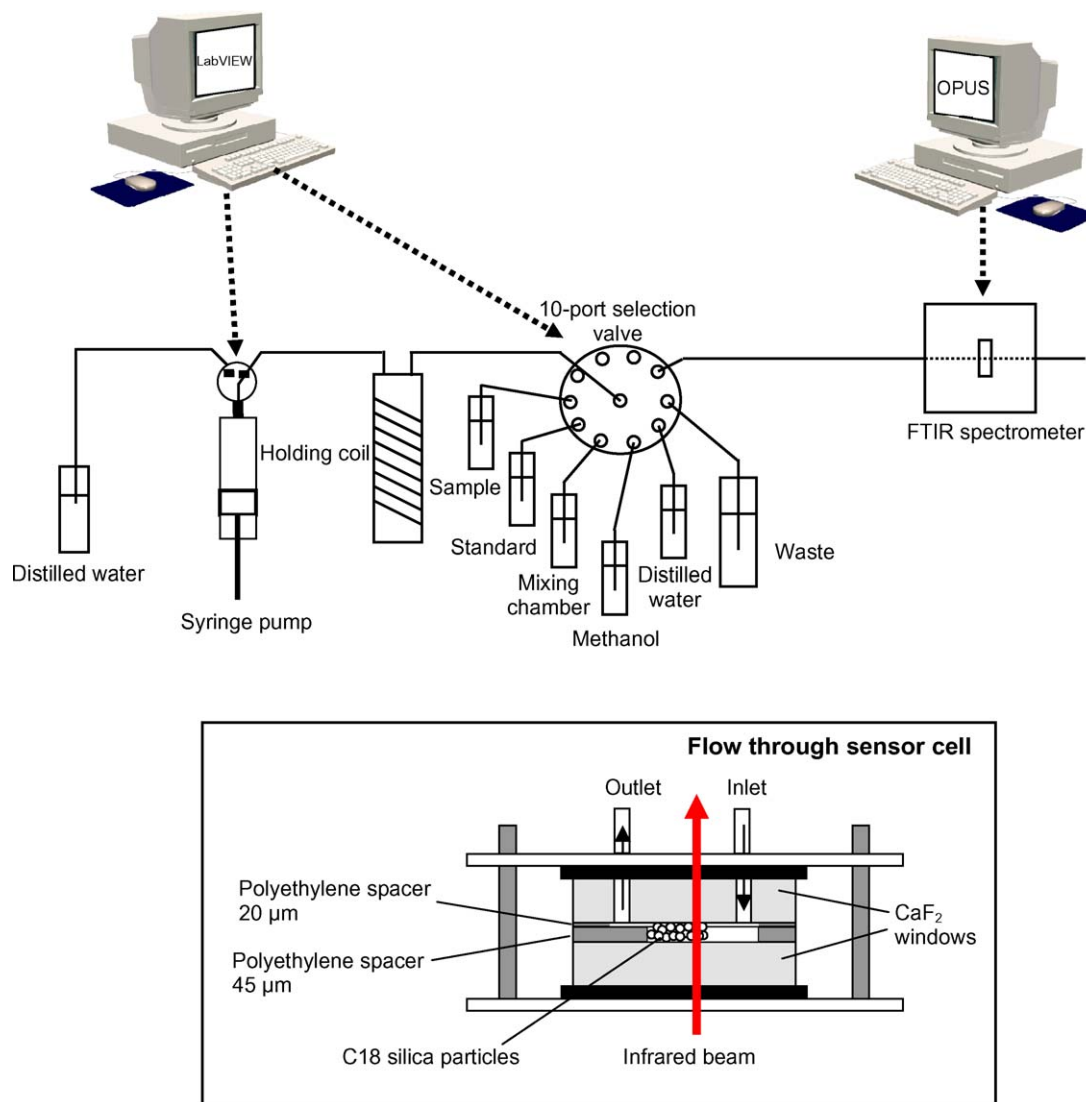
## 2. Experimental

### 2.1. Reagents and samples

All reagents were of analytical grade. Caffeine and methanol (HPLC grade) were supplied by Sigma (Schnelldorf, Germany). C18 BondElut sorbent material ( $56 \mu\text{m}$  average particle diameter) were purchased from Varian (Darmstadt, Germany). Soft drink samples were purchased from local markets and degassed in an ultrasonic water bath for 10 min before analysis. In order to evaluate the matrix effect on caffeine determination, normal cola, sugar-free cola and energy drink samples were purchase and analyzed.

### 2.2. FTIR instrument and sequential injection (SI) system

The automated manifold used is shown in Fig. 1. It comprises a Cavro (Sunnyvale, CA, USA) XP 3000 syringe pump (syringe size



**Fig. 1.** Scheme of the experimental setup consisting of an automated SI system and a mid-IR flow through cell equipped with two polyethylene spacers and containing C18 silica beads.

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