



## Cross injection analysis: Concept and operation for simultaneous injection of sample and reagents in flow analysis

Duangjai Nacapricha<sup>a,b,\*</sup>, Patidta Sastranurak<sup>a,b</sup>, Thitirat Mantim<sup>a,b</sup>, Natchanon Amornthammarong<sup>a,c</sup>, Kanchana Uraisin<sup>a,b</sup>, Chatchalida Boonpanaid<sup>a,d</sup>, Chatpong Chuyprasartwattana<sup>a,d</sup>, Prapin Wilairat<sup>a,b,e</sup>

<sup>a</sup> Flow Innovation–Research for Science and Technology Laboratories (FIRST Labs), Thailand

<sup>b</sup> Department of Chemistry and Center of Excellence for Innovation in Chemistry, Faculty of Science, Mahidol University, Bangkok 10400, Thailand

<sup>c</sup> National Oceanic and Atmospheric Administration (NOAA), Ocean Chemistry Division/AOML, Miami, FL 33149, USA

<sup>d</sup> Department of Industrial Chemistry, Faculty of Applied Science, King Mongkut's Institute of Technology North Bangkok, Bangkok 10800, Thailand

<sup>e</sup> National Doping Control Center, Mahidol University, Bangkok 10400, Thailand

### ARTICLE INFO

#### Article history:

Received 5 January 2013

Received in revised form

6 February 2013

Accepted 6 February 2013

Available online 13 February 2013

#### Keywords:

Cross injection analysis (CIA)

Simultaneous flow

Intersection zones

x-axis channel

y-axis channel

Iron

### ABSTRACT

This work presents a new flow injection strategy, called 'cross injection analysis' or CIA, an alternative cost-effective approach in flow analysis. The flow platform is made from a rectangular acrylic block, approximately  $5 \times 3 \times 1.5$  cm ( $x \times y \times z$ ), with crossing cylindrical channels drilled out along the  $x$ - and  $y$ -axis of the block. The outlet from the single  $x$ -axis channel is connected to a detector flow cell. This channel is filled with the carrier solution. The flow in the  $x$ -axis channel is driven by a computer controlled single-channel peristaltic pump. The multiple  $y$ -axis channels, running perpendicular to the  $x$ -channel, are connected to a multi-channel peristaltic pump. These channels contain the sample and reagent solutions that flow across the intersection zones of the channels. To mix the sample and reagent with subsequent detection of the reaction zone, flow is applied along the  $x$ -axis channel, while flow in the  $y$ -axis channels is stopped. We successfully demonstrated the validity of the CIA technique by the spectrometric determination of Fe(II) using 1,10-phenanthroline and the speciation of Fe(II) and Fe(III). To place the CIA technique within the context of flow analysis, a brief overview of the evolution of flow injection analysis and its later innovative development is included.

© 2013 Elsevier B.V. All rights reserved.

## 1. Introduction

Following the first report of flow injection analysis (FIA or FI) in 1975 by Ruzicka and Hansen [1], there have been a tremendous number of research papers published on new developments in this area [2–4]. FI can also be operated using the so-called 'reversed-FI' (rFI) [5,6], in which the reagent is injected into a flowing stream of the sample to improve the sensitivity over normal FI mode [7]. In 1990, Ruzicka and Marshall introduced sequential injection analysis (SIA or SI), the second generation of FI, that is fully controlled by the computer [8]. In SI technique, metered amounts of sample and reagent(s) are sequentially aspirated, through precise control of selection valve and the syringe pump (plunger in filling mode), as stacked layers into a holding coil. Finally, the reaction zone is propelled (plunger in delivering mode) to the detector. Ruzicka also proposed the lab-

on-valve (LOV) technique that extends the feature of sequential injection to provide liquid handling in microliter and sub-microliter levels of sample and reagent(s) with a microfluidic platform made of acrylic or other polymeric material attached atop the selection valve [9–11]. The microfluidic platform accommodates connecting ports, working channels and flow through detection cell. The LOV technique is often called SI-LOV or  $\mu$ SI-LOV [12]. In 2004, Grudpan proposed an alternative cost-effective approach to SI called lab-at-valve (LAV) [13]. Liquid handling in LAV utilizes a syringe pump similar to the SI concept. However in LAV, there is no replacement of the stator plate of selection valve by the precisely machined microfluidic-platform. Instead devices such as in-house potentiometric flow cell [14] or pipette tip, as SI liquid-liquid separator [15], are attached or plugged directly onto the port(s) of the SI selection valve.

Cerdà et al. proposed 'multi-syringe' (also written as 'multi-syringe') flow analysis or MSFIA in 1999 [16]. The basic system of MSFIA comprises four syringes that are connected in a block to the same stepping motor. A three-way solenoid valve is fitted at the head of each syringe for selecting the flow path (reservoir or analytical flow path). These valves are commutation valves that can be separately controlled without having to stop the

\* Corresponding author at: Department of Chemistry and Center of Excellence for Innovation in Chemistry, Faculty of Science, Mahidol University, Bangkok 10400, Thailand. Tel.: +66 2 2015122; fax: +66 2 2015127.

E-mail addresses: [duangjai.nac@mahidol.ac.th](mailto:duangjai.nac@mahidol.ac.th), [dnacapricha@gmail.com](mailto:dnacapricha@gmail.com) (D. Nacapricha).

movement of the syringe plungers. MSFIA is a versatile technique and the basic system can be modified to add on other components, such as solenoid valve(s) for sample introduction to eliminate carryover of sample in the syringe barrel [17].

Reis et al. introduced 'multicommutation' flow analysis (MCFA) [18]. MCFA is a continuous flow strategy that utilizes computerized control of three-way solenoid valves and a peristaltic pump to insert sample and reagent plugs into the liquid flow path. In the arrangement of the MCFA system, the pump is placed at the end of the flow line after the detection cell and only a single pumping channel is used. Later in 2002, Santos and others from the same laboratory presented another strategy called 'multi-pumping' (also written as multipumping) flow system (MPFA) [19]. In MPFA, a set of solenoid micropumps are individually controlled by a computer to propel sample and reagent(s) by time-based or a pulse counting approach. With suitable arrangement of micropumps, use of solenoid valves and peristaltic pump is thereby eliminated.

Recently in 2010, a group in Japan [20] presented a hybrid system of FI, SI and multicommutation called 'simultaneous injection-effective mixing analysis' (SIEMA). A SIEMA system consists of a syringe pump as the liquid delivery module. One part of the three-way solenoid valve fitted to the syringe head is connected to the analytical flow path by a four-way cross-connector. The system also has separately controlled three-way solenoid valves for introducing sample and reagents into the central flow line when the syringe plunger is pulled down (reverse flow). The reaction solution is later pushed into and out of the mixing coil for detection (forward flow).

In this work, we present a new flow analysis technique called 'cross injection analysis' or CIA. The CIA platform has one channel as the analytical flow path (the *x*-axis channel) and four channels (*y*-axis channels) perpendicular to this channel. These channels are drilled out of an acrylic rectangular block. Both ends of the *y*-axis channels are connected with tubings to the same set of rollers of a peristaltic pump to form four flow lines. In CIA, the sample and reagents are introduced into the analytical flow path of the platform (*x*-axis channel) via individual pump tube connected to the *y*-channels of the platform. Unlike the SI and the multicommutation techniques, liquid introduction in CIA is simultaneous. We employ the cross-flow mode to improve the mixing to achieve desired sensitivity. Although CIA may be similar to the SI and the multicommutation in the way that liquid zones are inserted into analytical flow path of the system, the pattern of liquid flow in CIA is different to these previous techniques due to the platform design and the employed cross-flow. We exploit the characteristic of the peristaltic pump in which rollers are in 'press' mode when the pump is stopped. Thus pressure is maintained during the stopped-flow period holding the liquid inside the tubing or connected channels without the need to use syringe pump or solenoid valve as in the SI or the multicommutation techniques, respectively. In this design, using a peristaltic pump, with eight pump tubes placed on the rollers, is equivalent to operating a set of eight solenoid valves.

## 2. Experimental

### 2.1. Reagents and samples

All chemicals used in this work were AR grade. Deionized-distilled water was used throughout all the experiments. Standard stock solution of Fe(II) ( $1,000 \text{ mg L}^{-1}$ ) was prepared by dissolving an accurate weight of 4.98 g iron (II) sulphate heptahydrate (Fluka, Switzerland) in 1.00 L of  $0.05 \text{ mol L}^{-1}$  sulfuric acid. Standard stock solution of Fe(III) ( $1000 \text{ mg L}^{-1}$ ) was prepared by dissolving an accurate weight of 4.71 g iron(III) sulfate heptahydrate (Sigma-Aldrich, USA) in 1.00 L of  $0.05 \text{ mol L}^{-1}$

sulfuric acid. These stock solutions were calibrated against a certified atomic absorption standard ( $1002 \pm 2 \text{ mg L}^{-1} \text{ Fe}$ ) as  $\text{Fe}(\text{NO}_3)_3$  in  $0.5 \text{ mol L}^{-1} \text{ HNO}_3$  (Merck, Germany) for accurate concentrations of iron. Working standard solutions of Fe(II) and Fe(III) were freshly prepared daily from the stock solutions by serial dilutions with  $0.05 \text{ mol L}^{-1}$  sulfuric acid. Acetate buffer pH 5.3, used as the carrier in the CIA system, was prepared by mixing 81.50 mL of  $2.00 \text{ mol L}^{-1}$  aqueous sodium acetate solution with 18.5 mL of  $2.00 \text{ mol L}^{-1}$  acetic acid in a one liter volumetric flask and making up to mark with water. The hydroxylamine solution was prepared by dissolving 19.8 g of hydroxylamine hydrochloride (Merck, Germany) in 250 mL of water. This solution was used to reduce iron (III) to iron (II) in the determination of total iron in the speciation analysis. The reagent for Fe(II) was 1,10-phenanthroline (Merck, Germany) in acetate buffer.

Five commercial products of multivitamin tablets were used in the validation study. Finely ground sample was weighed accurately (0.1 to 0.5 g) and dissolved in 100.0 mL of  $0.05 \text{ mol L}^{-1}$  sulfuric acid. After constant stirring for 30 min, the solution was centrifuged for 15 min at 3000 rpm. The supernatant was then filtered through a cellulose acetate membrane filter ( $0.45 \mu\text{m}$ ). The filtrate was directly aspirated into the CIA system.

### 2.2. CIA platform

Fig. 1a is a photograph of a CIA platform, which is made from Perspex™. Fig. 1b shows the dimensions of a regular platform with cylindrical channels drilled out using computer controlled drilling bit. The CIA platform has a main channel, designated as the '*x*-channel', for the carrier stream. The crossing channels (labeled 1–4 in Fig. 1c) are drilled perpendicular to the *x*-channel. These channels are designated as '*y*-channels' and are used for the sample and reagents solution. We can reduce or add *y*-channels according to the number of reagents required. For determination of iron, we used the CIA platform as shown in Fig. 1 with four *y*-channels.

### 2.3. The manifold

Fig. 2 is a schematic diagram of the CIA flow-manifold employed for the determination of iron (II) and total iron. An Ismatec peristaltic pump (model ISM843, Switzerland), designated as P1 with capacity for accommodating eight pump tubes, was used for simultaneously filling or withdrawing the sample/standard (S) and the three reagents (R1, R2 and R3), in the *y*-channels (channels 1 to 4) of the CIA platform. P2 is a peristaltic pump used for driving the carrier solution (C) through the *x*-channel (designated as channel 5 in Fig. 1c and in Fig. 2). For this work, we employed an Ismatec pump (model IS7610, Switzerland). The system was also equipped with an Upchurch valve, V (model V-101L, USA). When the valve is set at the 'open' position (solid line), the flow line of the CIA platform in the *x*-direction is connected to the detection cell of the detector D, and then to waste (waste 2). When the valve V is set to 'close' position (dashed line), the carrier solution flows out of the platform via the *y*-channels to waste 1. A Jenway spectrophotometer (model 6450, UK) with 1-cm path length flow-through cell (Hellma, USA) was employed for absorbance measurement at 510 nm. Labview™ program, version 8.0, was used for data acquisition of the signal from the detector. An in-house CIA electronic board was built to control the peristaltic pumps P1, P2 and the valve V using software written in Visual Basic 6.0.

### 2.4. Investigated operating procedures

In this work, there are two operating procedures that were investigated as shown in Table 1. In procedure 1, flow in the

Download English Version:

<https://daneshyari.com/en/article/7682506>

Download Persian Version:

<https://daneshyari.com/article/7682506>

[Daneshyari.com](https://daneshyari.com)