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Development and application of novel flow method – Stepwise injection analysis

Several flow methods with forced convection such as Zone fluidics in flow analysis (ZF) [1], Flow batch analysis (FBA) [2], Sequential injection analysis with a mixing chamber (SIA MC) [3], Stepwise injection analysis (SWIA) [4] and Multicommutated Stepwise injection analysis (MCSWIA) [5] have been developed for an automation of chemical analysis. These methods include mixing the solutions in a mixing chamber (MC) to achieve physical and chemical equilibrium and high sensitivity.

In a certain sense, ZF can be considered as a return to a concept of Segmented flow analysis, but using the experience gained in the evolution of Sequential injection analysis (SIA). In ZF portion of «fluid» (an immiscible with the sample flow phase – gas or liquid), the sample, a solution of reagents and again «fluid» are delivered using a reversible pump into a holding coil through a valve. After that, an isolated segment of the sample is transfered into a sample pre-treatment device, which can function as the MC. At the final stage, solution from this pre-treatment device is delivered to the detector.

In FBA portions of the sample and reagents solutions using several peristaltic pumps are delivered into the MC. Mixing the solutions is carried out in the MC with a magnetic stirrer. After mixing the solutions into the MC, a pause is made to complete the chemical reaction and the measurement of an analytical signal is performed. Nevertheless, the FBA manifold proposed by the authors has certain limitations. The combination of the MC with the detector limits the possibility of varying the sample volume. The increasing of the optical path length for absorbance measuring is less possible.

The SIA MC manifold differs from FBA in the conditions of the sample zone formation. Mixing of the sample with the reagent solutions is carried out in the MC, but after there, the solution is injected into a carrier solution and delivered through the reaction coil to a flow detector. This manifold is most similar to SIA, but it eliminates the problem associated with an inefficient mixing the sample zone and reagent solutions as in SIA. However, when delivering the reaction product to a detector in the carrier solution, the dispersion of its zone is observed and the sensitivity of the method is decreased.

In our work, a new concept for the automation of chemical analysis has been presented. The concept of SWIA assumes that all stages of routine analysis are performed strictly: sampling; sample preparation, including concentration and separation; effectively mixing solutions by a gas flow; thermostating; and finally the measurement of the analytical signal. The SWIA manifold includes a multi-way valve, a reversible peristaltic pump, a flow detector, a thermostated MC, a gas delivering channel for mixing the sample and reagent solution in the MC or for the dissolution of solid-phase samples, liquid-liquid extraction and absorption of gases. The SWIA does not require special devices for mixing solutions and allows automating of solid-phase samples dissolution and the absorption of gases.

Later the MCSWIA was proposed for an automation of a multicomponent spectrophotometric analysis. The MCSWIA manifold includes two similar solenoid valves and two peristaltic pumps. The first valve is used for sequential injection of samples, reagent solutions and a gas phase into the flow system. The gas phase is used for mixing of solutions in the MCs which are input to the second valve. The number of MCs is determined by the number of analytes and the corresponding number of colour-forming reactions, which are necessary for their determination. In turn, the number of MCs is limited by the number of ports of a valve.

During the last eight years the SWIA has been explored successful to automate various analytical procedures including the dissolution of solid-phase samples [6, 7], the liquid-liquid [8] extraction and the absorption of gases [9, 10].

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