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The development of flow injection analysis incorporating membraneless separation of volatile compounds and novel designs of vaporization unit

Most of analytical procedures require at least one separation step for sample preparation before measurement. On-line sample treatment in FIA and SIA provides many advantages in terms of high sample throughput, simple automation system, low sample consumption and reduce risks of contamination in a close system. With volatile compounds, on-line gas-liquid separation can be incorporated into the flow system to facilitate sample clean up or for selective detection. Conventional diffusion device for on-line separation is the gas diffusion unit (GD) [1]. GD is used in FIA to transfer a gaseous compound from one solution stream to another. The two streams, which are denoted as donor and acceptor are separated by a permeable hydrophobic membrane. In this way, selective detection of the diffused volatile analyte is achieved. However, using membrane, GD is not cost effective and has risk of contamination and clogging.

In 2006, an innovative membraneless gas diffusion unit (MGD) was presented by our group for the separation and analysis of volatile compounds [2]. The unit consists of donor and acceptor channels parallel to each other separated by a thin wall. The channels are connected by the headspace above the chambers where diffusion of the gaseous analyte occurs. Determination of ethanol in alcoholic beverages was carried out to demonstrate the potential application of this new concept of non membrane gas separation. It was found that the MGD unit provided a greater mass transfer than the GD unit. Another design with slight modification was presented for analysis of petrol [3]. The unit can be opened to clear residual vapor in the head space at the end of each measurement.

A non-membrane apparatus was also independently reported by Mornane et al. called thin layer distillation [4]. The apparatus was tested in a flow-based format for the determination of ammonia and amines. Later on, Ines et al. modified the MBL unit to improve our design [5]. The re-designed unit having a 'bath tub' form, coupled with a multi-syringe flow analysis system, was explored for its potential use. Enhancement of sensitivity was attained by increasing the temperature of the unit by means of a thermostating water stream flowing underneath the donor channel.

It should be noted that all previous designs required equal flow rate of inlet and outlet stream to avoid overflow and cross contamination (by flooding) between the channels, which sometimes is difficult to control. In 2013, we presented the development of a new design for membraneless vaporization (MBL-VP) unit, called dual chambers- MBL-VP [6]. With this design, exact volumes of sample and reagent are introduced into their respective cone-shaped chambers from the base of the cone. Diffusion of volatile analyte takes place in the headspace above the dual chambers. After an appropriate time interval, acceptor solution is withdrawn from the same port at the base of chamber into the detector, while the sample solution is withdrawn to waste. Unlike the previous design, problems with overflow of solutions are completely eliminated. In addition, there is a small hole above the top level of the chambers to prevent excessive build up of pressure inside the unit. The newly design unit is also capable of accelerating the gas diffusion process by continuous pumping of air into the sample solution in the donor chamber. As the result, relatively short analysis time of 140 s was achieved for ethanol content in a range of 5 %v/v to 50 %v/v. Percent recovery was in the range of 96 % to 109%. The MBL-VP unit is being applied to the simultaneous measurement of more than one volatile compound. Further modifications and optimizations of the system are being investigated.

References

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