

---

# DEVELOPMENT OF A PERISTALTIC PUMP ASSISTED DYNAMIC HEADSPACE SAMPLING TECHNIQUE FOR NEEDLE TRAP DEVICE FOLLOWED BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY

Esmaeil Babanezhad, Faezeh khalilian, Mehrnoush Naderi and Habib Bagheri

Department of Chemistry, Sharif University of Technology, Tehran, Iran,  
bagheri@sharif.edu

In the last few years, the inside needle capillary adsorption trap (INCAT) technique as a solventless sample preparation approach and alternative extraction method derived from SPME has been developed [1,2]. This technique can be placed into one of the two categories based on which approach is used; one is the sorbent placed as adsorbent layer, acting as non-polar and polar phase, on the interior surface of needle [3]. The other category is in-needle packing with commercially available sorbents or chemically synthesized polymers [4]. An aniline/o-phenylenediamine copolymer was chemically synthesized and used as a sorbent material for needle trap device. A peristaltic pump was employed for dynamic headspace sampling of chlorobenzenes from aqueous samples. The extractant was subsequently into a conventional GC injector.

The porous surface structure of the coating was examined using the scanning electron microscopy (SEM). Possessing porous structure which provides higher specific surface area and loading capacity, capability of the chemical structure of sorbent for contribution in  $\pi$ - $\pi$  interaction between analytes and the sorbent makes the needle trap device a rather sensitive for extracting aromatic compounds.

The experimental parameters as breakthrough volume, linearity, repeatability and LOD were investigated. Using a small amount of sorbent particles, 1.4 mL/min was the highest sampling flow rate that could be used with no breakthrough of any chlorobenzenes components. LOD ranges from 0.005 to 0.010 ng/mL and %RSD ranges from 6.5% to 11% at concentration 0.1 ng/mL. The performance of sorbent was, also, compared with a commercial SPME fiber, polydimethylsiloxane/divinylbenzene, under similar experimental conditions. The proposed method was successfully applied to the extraction of some selected chlorobenzenes from real-life water samples and the relative recoveries were higher than 90% for all the analytes.

## References:

- [1] J.A. Koziel, M. Odziemkowski, J. Pawliszyn, *Anal. Chem.* 73 (2001) 47.
- [2] A. Wang, F. Fang, J. Pawliszyn, *J. Chromatogr. A* 127 (2005) 1072.
- [3] I.Y. Eom, A.M. Tugulea, J. Pawliszyn, *J. Chromatogr. A* 3 (2008) 1196.
- [4] I.Y. Eom, V.H. Niri, J. Pawliszyn, *J. Chromatogr. A* 10 (2008) 1196.