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Liquid-EI interface: A new concept to enhance performance and stability in liquid chromatography-electron ionization mass spectrometry

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A novel liquid chromatography-mass spectrometry (LC-MS) interfacing concept is presented and discussed. The new interface design is called "Liquid-EI", LEI to distinguish it from previous attempts. In a LEI interface, vaporization of the HPLC eluate is carried out inside a suitable, independent micro-channel right before entering the ion source. An inert gas flow carries the gas phase molecules into the ion source. This approach moves the solute vaporization event immediately outside the ion source into a more suitable space in terms of dimensions, temperatures and surface materials, and free of sensitive components and electric potentials. The pressure drop and temperature gradients between LC and MS can be carefully monitored and controlled to enhance analytes response and reduce band broadening and/or solute carryovers. Preliminary results carried out using an Agilent 7010 QqQ mass spectrometer and 5975 single quadrupole, gave us an optimistic impression, especially when the vaporization micro-channel was covered with a ceramic layer. Proof of concept and detailed description of the interface are presented. Preliminary experiments were conducted using PAHs (2-6 rings) and other compounds of environmental interest with and without column separation. Real world samples were analyzed using LEI with particular emphasis to address issues on identification of metabolites at low levels where mass spectral quality using high resolution accurate mass (HRAM) can be an issue and NMR may prove challenging, and further, complex sample matrix (soil, vegetable, food and other bio matrices, etc..) can give additional difficulties.

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Combination of SEC & MALDI techniques for characterization of the continuous phase in dispersion polymerization

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Dispersion polymerization is one of the most attractive dispersed phase polymerization techniques to synthesize nano- and microparticles, since it allows the synthesis of particles with narrow and broad size distributions from a single polymerization step. Dispersions of latex particles in polar and non-polar solvents are important materials in several areas, such as coatings, toners, column packing materials for chromatography, printing plates for lithography, sensors for biomedical and biochemical analysis. Most of the works carried out to understand this type of polymerizations mechanism are related to the study of stabilizer type and concentration, continuous phase composition and type of comonomer. Nevertheless, there are few works related to the characterization of the polymers formed in the continuous phase (also known as the serum). In this work, the dispersion copolymerization serum of styrene (St) with two different comonomers acrylonitrile (AN) and methyl methacrylate (MMA) in 1-propanol/DDI water medium carried out using polyethylene oxide macro-monomer as stabilizer was studied. The serums of the latexes were characterized in terms of molar mass and composition by the combination of Size Exclusion Chromatography (SEC) and Matrix Assisted Laser Desorption Ionization Time of Flight Mass Spectrometry (MALDI-TOF MS). It was found that unreacted macro-monomer was present in both copolymer systems. However, in the case of the copolymerization with AN besides the presence of the macro-monomer, there were also some pAN oligomers. This work demonstrates that the combination of SEC-MALDI techniques can be a powerful strategy for identifying the different species present in the continuous phase in dispersion polymerization.

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