Ion Mobility Spectrometry and High Resolution Mass Spectrometry for the Characterization of Extremely Air Sensitive Metallocene Catalysts

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Metallocenes are extremely air sensitive catalysts that are used in the production of polyolefins¹ and they are difficult to characterize.² NMR is a routine technique used for characterization of metallocenes but it has some limitations to paramagnetic metals and complex mixtures. Not only metallocenes are air sensitive but also they are sensitive to nucleophiles, so chromatography techniques (such as GC-MS, LC-MS) cannot be applied to purify and analyze them. Conversely, mass spectrometry allows the separation and identification of complex mixtures with no limitation to their electronic configuration states. We have been developed so far the ability to use a source called ASAP (Atmospheric Solids Analysis Probe)³ to analyze highly air sensitive metallocenes. This technique is based on depositing the sample on the tip of a capillary tube and introducing it into ASAP source, in which the sample is desorped by hot N₂ and ionized by nitrogen plasma generated by a corona discharge. We have modified this technique toward new simple approach, referred to as inert ASAP (Figure 1). In this course, we would like to present recent results on the analysis of extremely air sensitive metallocenes by inert ASAP mass spectrometry coupled to ion mobility, that leads to a bidimentional separation in the gaseous phase. We highly believe that this technique can be extended for the analysis of other interesting very air sensitive complexes that are used in wide applications which cannot be analyzed by other analytical techniques.

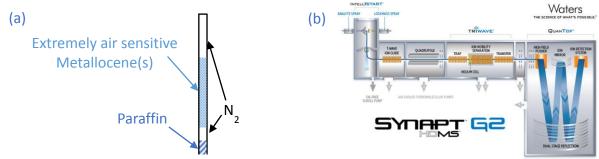


Figure 1. (a) inert ASAP protocole. (b) Synapt G2 (QTOF mass spectrometer, Waters) used for the analysis of air sensitive metallocenes.

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²⁾ P. M. MORSE, Chem. Eng. News Archive, 1998, 76, 25.

³⁾ C. N. McEwen, R. G. McKay and B. S. Larsen, Anal. Chem., 2005, 77, 782