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Mass Spectrometry Congress 2019: High-performance liquid chromatography mass spectrometry in the study of small metal clusters - Yuichi Negishi - Tokyo University of Science, Japan

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Small metal clusters have attracted considerable attention as new functional nanomaterials because they have size-specific properties and functions that are not found for corresponding bulk metal. In particular, hydrophilic thiolate-protected gold clusters (hereinafter referred to as hydrophilic gold clusters) exhibit high biocompatibility and luminescence quantum yield in addition to pollution-free properties. Therefore, hydrophilic gold clusters are expected to be used in biomedical and environmental applications. Replacing some of the Au atoms in these clusters with different elements may impart them with even more useful functions. However, the synthesis of hydrophilic metal clusters has been less studied because of the complexity involved in evaluating the mass distributions of product mixtures. In this work, we found two hydrophilic interaction liquid chromatography (HILIC) columns for high-performance liquid chromatography (HPLC) suitable for the high-resolution separation of hydrophilic metal clusters. The mass distributions of the product mixtures of hydrophilic metal clusters were evaluated via HPLC mass spectrometry (LC/MS) using these HILIC columns. Consequently, we observed multiple clusters that had not been previously reported for glutathionate (SG)-protected gold clusters (AuN(SG)M). Additionally, author demonstrated that Aun−xMx(SG)M alloy clusters (M = Ag, Cu or Pd) in which part of the Au in the AuN(SG)M cluster is replaced by a heteroelement can be synthesized, similar to the case of hydrophobic alloy clusters. It is easy to evaluate the mass distributions of hydrophilic metal clusters using this method. Thus, remarkable progress in the synthesis techniques of hydrophilic metal clusters through the use of this method is anticipated, as the situation for hydrophobic metal clusters.

Liquid chromatography is a method of physical separation. Between two immiscible phases, i.e. stationary and mobile, the components of a liquid mixture are distributed in the Liquid chromatography method. The practice of LC can be divided into five categories, namely adsorption chromatography, partition chromatography, ion exchange chromatography, size exclusion chromatography and affinity chromatography. Among these, the most widely used variant is the reverse phase (RP) mode of the partition chromatography technique, which uses a nonpolar (hydrophobic) stationary phase and a polar mobile phase. In common applications, the mobile phase is a mixture of water and other polar solvents (e.g., methanol, isopropanol, and acetonitrile), and the stationary matrix is prepared by attaching long chain alkyl groups (e.g., n -octadecyl or C18) on the surface of silica particles 5 μm in diameter of irregular or spherical shape.

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Mass spectrometry (MS) is an analytical technique that measures the mass / charge ratio (m / z) of charged particles (ions). Although there are many types of mass spectrometers, all of them use electric or magnetic fields to manipulate the movement of ions produced from an analyte of interest and determine their m / z. The basic components of a mass spectrometer are the ion source, mass analyzer, detector, and data and vacuum systems. The ion source is where the components of a sample fed into an MS system are ionized using electron beams, photon beams (UV lights), laser beams, or corona discharge. In the case of electrospray ionization, the ion source moves the ions that exist in the liquid solution into the gas phase. The ion source converts and fragments the neutral molecules in the sample into gas phase ions which are sent to the mass analyzer. While the mass analyzer applies electric and magnetic fields to sort ions by their masses, the detector measures and amplifies the ion current to calculate the abundances of each mass resolved ion.

Liquid Chromatography-Mass Spectrometry (LC-MS) technique combines liquid chromatography's physical separation capabilities with mass spectrometry's mass analysis capabilities. Coupled Chromatography - MS systems are popular in chemical analysis because the individual capabilities of each technique are synergistically enhanced. With high molecular specificity and detection sensitivity, mass spectrometry provides the structural identity of individual components while liquid chromatography separates mixtures with multiple components. For analyzing biochemical, organic and inorganic compounds that are usually found in complex samples of environmental and biological origin, this tandem technique can be utilized. Hence, in a wide range of industries including biotechnology, food processing etc., LC-MS can be applied.

In addition to liquid chromatography and mass spectrometry devices, an LC-MS system contains an interface that efficiently transfers the separated components from the LC column to the MS ion source. The interface is necessary because LC and MS devices are fundamentally incompatible. Thus, it is not possible to directly pump the eluate of the LC column in the MS source. The interface should not interfere with the ionization efficiency and vacuum conditions of the MS system. Today, the most widely applied LC-MS interfaces are based on atmospheric pressure ionization (API) strategies such as electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI) and atmospheric pressure photoionization (APPI).

The mass spectrum can be used to determine the mass of analytes, their elemental and isotopic composition, or to elucidate the chemical structure of the sample. MS is an experiment which must take place in gas phase and under vacuum (1.33 * 10−2 to 1.33 * 10−6 pascal). Therefore, the development of devices facilitating the transition of samples at higher pressure and in condensed phase (solid or liquid) in a vacuum system has been essential to develop MS as a powerful tool for the identification and quantification of organic compounds and peptides. MS is now very commonly used in analytical laboratories that study the physical, chemical or biological properties of

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a wide variety of compounds. Among the many types of mass analyzers, those which find application in LC-MS systems are quadrupole, time-of-flight (TOF), trap ion and quadrupole-TOF (QTOF) hybrid analyzers.

The interface between a liquid phase technique (HPLC) with a continuous flow eluate and a gas phase technique performed under vacuum has been difficult for a long time. The advent of electrospray ionization changed that. Currently, the most common LC-MS interfaces are electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI), and atmospheric pressure photoionization (APPI). These are new sources of MS ions that facilitate the transition from a high pressure environment (HPLC) to the high vacuum conditions required by the MS analyzer. Although these interfaces are described individually, they may also be commercially available as ESI / APCI, ESI / APPI or APCI / APPI dual ion sources. Various deposition and drying techniques have been used in the past (eg, moving belts), but the most common of these has been offline MALDI deposition. A new approach still under development called LC-MS direct-EI interface, combines a nano HPLC system and a mass spectrometer equipped with electronic ionization.

Biography:

Yuichi Negishi received his PhD Degree from Keio University, Japan in 2001. He is the Professor of Tokyo University of Science, Japan. He has over 150 publications that have been cited over 8,000 times. In his publications, 10 papers were categorized to Top 1% cited papers and 18 papers were selected as cover picture of the journal. His publication H-index is 45. He has been awarded several prizes, including the PCCP Prize in 2007, CSJ Award for Young Chemists in 2008, Japan Society of Molecular Science Award for Young Chemists in 2012 and Yagami Prize in 2017.

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