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Pharmacology & Chromatography & HPLC Congress 2018: Atomic and isomeric high-resolution separation of thiolate-protected alloy clusters by reversed-phase high-performance liquid chromatography - Sayaka Hashimoto - Tokyo University of Science

Sayaka Hashimoto[1] and Yuichi Negishi[2]
Tokyo University of Science, Japan

By doping heteroatoms to thiolate-protected gold clusters, we can add more functionality to the cluster depending on the number of doped heteroatoms and the difference in doping positions. However, in the synthesis of alloy clusters, the mixture of clusters with a different number of heteroatoms is produced. Therefore, a precise separation of each cluster is required to understand their properties. In this study, we attempted to separate gold-silver alloy clusters precisely, furthermore investigated isomer change by reversed-phase high-performance liquid chromatography (RP-HPLC). Au38-nAgn(SC4H9)24 was used as a sample. This cluster was synthesized by two methods, (1) by adding [Ag(SC4H9)] complex to Au38(SC4H9)24 (metal exchange) and gold and silver ions were reduced in the presence of butanethiol in solution (co-reduction). The mixture of alloy clusters was separated by RP-HPLC using gradient program for controlling mobile phase, and core-shell type column. The peaks obtained from the chromatogram were evaluated by electrospray ionization (ESI) mass spectrometry connected to RP-HPLC directly. Figure.1 (a) shows the chromatogram of Au38-nAgn(SC4H9)24 obtained by the metal exchange. Some clear peaks were observed in chromatogram. Each peak was attributed to the cluster having a precise number of silver atoms (Figure. 1(b)). These results indicate that the mixture of Au38-nAgn(SC4H9)24 was precisely separated according to the number of silver atom. Furthermore, the shape of chromatogram of Au38-nAgn(SC4H9)24 prepared by the metal exchange changed by leaving this cluster in toluene for 6 days (Figure. 1(c)). Interestingly, the shape was similar to that of Au38-nAgn(SC4H9)24 prepared by the co-reduction (Figure. 1 (d)). These results suggest that Au38-nAgn(SC4H9)24 prepared by the metal exchange contains metastable clusters, these are transformed to the stable clusters by leaving in toluene. In conclusion, we have succeeded in the high-resolution separation of alloy clusters according to each chemical composition, and observation of isomer transformation.

Recent Publications:

1. Y Negishi, et al. (2016) Precise synthesis, functionalization and application of thiolate-protected gold clusters. Coord. Chem. Rev., 320-321:238-250.
2. Y Negishi, et al. (2016) High-resolution separation of thiolate-protected gold clusters by reversed-phase high-performance liquid chromatography. Phys. Chem. Chem. Phys. (Perspective) 18:4251-4265.
3. Y Negishi, et al. (2015) Understanding ligand-exchange reactions on thiolate-protected gold clusters by probing isomer distributions using reversed-phase high-performance liquid chromatography. ACS Nano 9: 9347-9356.
4. Y. Negishi, et al. (2015) A critical size for emergence of nonbulk electronic and geometric structures in dodecanethiolateprotected au clusters. J. Am. Chem. Soc. 137:1206-1212.
5. Y. Negishi, et al. (2013) Separation of precise compositions of noble metal clusters protected with mixed ligands□J. Am. Chem. Soc. 135:4946-4949.