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Precise separation of gold clusters protected by hydrophilic thiolates and evaluation of their chemical compositions by LC/MSKana Yoshida and Yuichi Negishi
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Small thiolate-protected gold clusters show size-specific physical and chemical properties, which are not observed in bulk gold. Especially, hydrophilic thiolate-protected gold clusters exhibit a good affinity for biomaterials. Therefore, much research has been conducted in this field. However, it is difficult to selectively synthesize the clusters with specific chemical composition. Therefore, to evaluate properties of hydrophilic thiolate-protected gold clusters accurately, we need to separate single cluster from the mixture of these clusters with high resolution. In this work, we report on precise separation of various hydrophilic thiolate-protected gold clusters (Au_n(SR)_m, SR=SG (glutathionate), NALC (N-acetyl-L-cysteine), p-MBA (p-mercaptobenzoic acid)) by hydrophilic interaction liquid chromatography (HILIC). Furthermore, we attempted to evaluate the chemical composition of each cluster by introducing LC/MS which was directly connected the chromatograph with the mass spectrometer. The mixture of clusters used in this work was synthesized by reducing gold ions in the presence of thiols. Figure 1(a) shows the UV chromatogram of Au_n(SG)_m. Multiple peaks were observed in the chromatogram. Figure 1(b) shows the ESI-mass spectrum of each peak observed in the chromatogram. It was found that almost only one cluster was contained in each peak. These results indicate that mixture was separated with high resolution according to the chemical composition of clusters. Similar separation has also been achieved for Au_n(NALC)_m and Au_n(p-MBA)_m. Therefore, it was revealed that the use of HILIC columns is powerful tool for separating of gold clusters protected by many kinds of hydrophilic thiolates. Furthermore, Although chemical compositions observed for Au_n(SG)_m and Au_n(NALC)_m were similar each other, that for Au_n(p-MBA)_m was less compared with these clusters. These results indicate that chemical compositions of the Au_n(SR)_m vary depending on the ligand structure. In this manner, we have succeeded in revealing how difference of ligand affects synthesis of gold clusters protected by hydrophilic thiolate ligands.

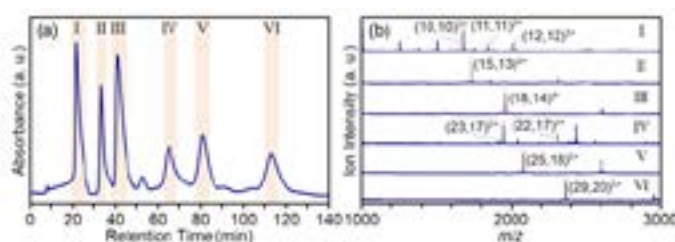


Figure 1: (a) UV chromatogram of Au_n(SG)_m. (b) Mass spectra of each peak (I-VI). The notation (n, m)^{z+} indicates [Au_n(SG)_m-zH]^{z+} for n = 10-12, 15, 18, and 23, whereas it indicates [Au_n(SG)_m+z]^{z+} for n = 22, 25, and 29 because of the negative charge of the metal cores of Au₂₂(SG)₁₇, Au₂₅(SG)₁₈, and Au₂₉(SG)₂₀.

Figure 1: (a) Chromatograms obtained by metal exchange, (b) ESI spectrum of each peak in chromatogram, (c) Chromatograms obtained by metal exchange after 6 days, and (d) Chromatograms obtained by co-reduction.

Recent Publications

1. K Yoshida, Y Negishi, et al. (2018) High-performance liquid chromatography mass spectrometry of gold and alloy clusters protected by hydrophilic thiolates. *Nanoscale*, 10:1641-1649.
2. Y Negishi, et al. (2017) Separation of glutathionate-protected gold clusters by reversed-phase ion pair high-performance liquid chromatography. *Ind. Eng. Chem. Res.* 56:1029-1035.
3. Y Negishi, et al. (2016) Precise synthesis, functionalization and application of thiolate-protected gold clusters. *Coord. Chem. Rev.* 320-32:238-250.

4. Y Negishi, et al. (2015) A critical size for emergence of nonbulk electronic and geometric structures in dodecanethiolate-protected au clusters. *J. Am. Chem. Soc.* 137:1206-1212.
5. Y Negishi, et al. (2014) Advanced use of high-performance liquid chromatography for synthesis of controlled metal clusters. *Nanoscale* 6:7889-7896.

Biography

Kana Yoshida has completed her BSc in 2017 in Applied Chemistry at Tokyo University of Science, Japan. Her main research interests are high resolution separation of noble metal clusters protected by hydrophilic thiolate ligands. She has presented her work many times at conferences. Furthermore, her research was published to *Nanoscale* (IF=7.367) which is one of the high impact factor journal in nanoscience field.

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