

# Soft X-ray photoelectron spectroscopy of the metal complex included in cyclodextrin

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## Introduction

Gold is a finite resource and is currently used for decoration and inside electronic devices. In recent years, there has been an urgent need to recover and reuse gold contained in industrial waste. In this study, we focused on the galvanic substitution reaction as a method to recover gold from  $\text{AuBr}_4^-$ , which is formed by dissolving gold during the recovery process. The galvanic substitution reaction is a spontaneous reaction caused by the difference in standard redox potentials [1]. When a Ni plate is immersed in a  $\text{KAuBr}_4$  solution,  $\text{AuBr}_4^-$  undergoes galvanic substitution with Ni and Au is deposited. During this process, the reddish-brown solution turned transparent. In order to obtain information about the precipitates on Ni, photoelectron spectroscopy experiments were performed at BL7 in HiSOR, and the photoelectron spectra in the Au 4f region were discussed.

## Experiment

Ni discs (0.1 mm thick,  $\phi = 3$  mm) were immersed in 20 mM  $\text{KAuBr}_4$  solution in a vial to precipitate the Au complex; if CD was added, the equivalent of 40 mM was added. After immersing the Ni plate in each solution for more than 24 hours, the sample was removed onto filter paper, washed three times with milli-Q water, and transferred to a vacuum chamber to dry. The sample holder was made of oxygen-free copper and silver paste was used to bond the holder to the sample. To investigate the electronic structure of the deposited gold, photoelectron spectroscopy was performed at HiSOR BL-7 using SR in the incident light energy range from 50 eV to 400 eV.

## Results and Discussion

Figure 1 shows the soft x-ray photoelectron spectra in the Au 4f region of the precipitates. The incident light energy is 200 eV. Neutral Au ( $\text{Au}^0$ ; 0-valent) has 4f<sub>7/2</sub> and 4f<sub>5/2</sub> peaks at 83.8 and 87.1 eV, respectively. On the other hand, when ionized, each peak shifts to the higher binding energy side, with peaks at  $\text{Au}^{1+}$  (85.6 and 89.1 eV) and  $\text{Au}^{3+}$  (87.3 and 90.4 eV), respectively [2]. Gold bromide (present as  $\text{Au}_2\text{Br}_6$ ) is also stable as a neutral compound containing gold and bromine. It may precipitate and in this case,  $\text{Au}^{3+}$  would be observed. Comparison of the Au 4f photoelectron spectra of the fresh  $\text{Au}^0$  film already installed at BL-7 and the present precipitates on the Ni plate surface shows that the peak energies of the respective 4f peaks are almost same between them. It is, thus, indicating that the precipitated material is not  $\text{Au}_2\text{Br}_6$  but neutral gold  $\text{Au}^0$ . The peak position does not shift when cyclodextrin was added. These results confirm that the galvanic substitution reaction occurred under the present experimental conditions. In the future, we plan to obtain

information on the reaction rate by examining the time variation of the UV spectra, and to try to control the reaction by using inclusion by cyclodextrins.

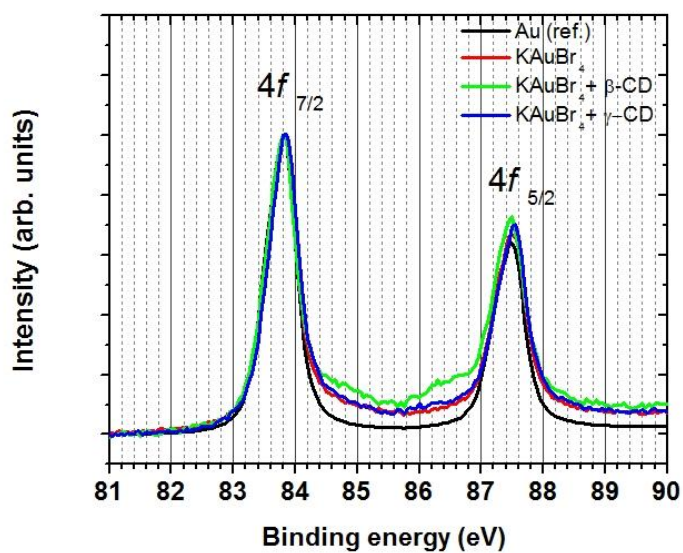


Figure 1 Comparison of the soft x-ray photoelectron spectra in the Au 4f region between a fresh Au film and the respective precipitates; KAuBr<sub>4</sub>, KAuBr<sub>4</sub>+β-CD, and KAuBr<sub>4</sub>+γ-CD films.

## REFERENCES

- [1] A. Volta, *Phil. Trans. Royal Soc.* **90** (1800) 403–431.
- [2] J.-P. Sylvestre et al., *J. Phys. Chem. B* **108** (2004) 16864-16869.