

数粒の陰イオン交換樹脂濃縮による金の定量法

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Determination of Gold by Pre-Concentration on an Anion Exchange Resin Bead

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Abstract

The analytical conditions of pre-concentrating Au on anion exchange beads and then stripping it from the beads into a $\text{H}_2\text{NCSNH}_2\text{-HCl}$ solution were examined. The reproducibility was $\pm 4\%$ at $100\ \mu\text{g Au/l}$ level while the recovery of Au was slightly low (85%). This method was used to measure the uptake rate of Au by marine phytoplankton.

Introduction

Trace metals, especially iron and manganese, are essentially important elements for the plankton growth. Generally, the concentration of trace metals in natural aquatic environments is in the level of a few to a hundred nanograms per liter. In analysis using anodic stripping voltammetry (i.e., Heggie, 1982; Bruland *et al.*, 1985) or cathodic stripping voltammetry (i.e., Van den Berg, 1984), pre-concentration of the metals are unnecessary due to their high sensitivity. In the case of flameless atomic absorption spectrophotometry (FAAS) now widely being used, the metals in a sample solution should be pre-concentrated prior to analyses. To carry out pre-concentration, the solvent extraction, chelex resin and coprecipitation methods are commonly used. It is difficult, however, to avoid metal contamination to the samples during pre-concentration except in the case of some metals.

Koide *et al.* (1984) measured Cd, Pd, Ir, Au and Pu in seawater by concentrating them on a few anion exchange resin beads. The beads were directly introduced into a carbon cup atomizer in FAAS. This method is excellent for avoiding metal contamination to the samples, however it did not result in good reproducibility due to the difficulty for introducing the beads into the atomizer.

In this paper, we tried stripping Au from the beads into a solution after Au absorption and furthermore examined the analytical conditions in the stripping step. This method is effective in separating Au in a salt solution prior to FAAS analysis.

Experiment

Reagents

All reagent solutions were prepared with doubly distilled water obtained by the use of a quartz glass stiller.

Potassium thiocyanate: Dissolve 2 g of KSCN in 100 ml of distilled water.

Thiourea: Dissolve 0.7 g of H_2NCSNH_2 in 1 ml of conc. HCl and dilute to 100

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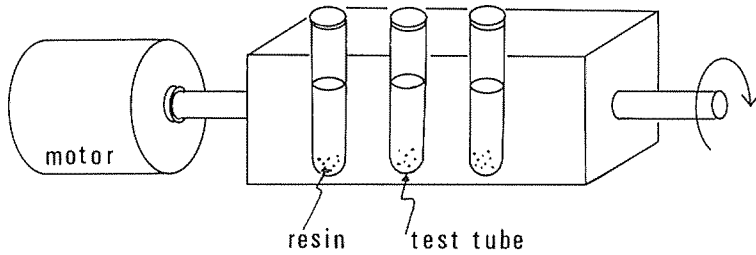


Fig. 1. Apparatus for rotating the resin beads.

ml with distilled water.

Au standard solution : An Au standard solution for atomic absorption spectrophotometry was employed (Wako Chemical Co. LTD.).

Resin : Amberlite IRA-900 (Sigma Chemical Co. LTD.) with a diameter of 0.5 mm was employed. The resin was cleaned by repeatedly soaking in 3M HNO₃ and 3M HCl.

Apparatus

The apparatus for rotating the resin beads consists of an ac moter, speed controller and a cylindrical drum (Fig. 1).

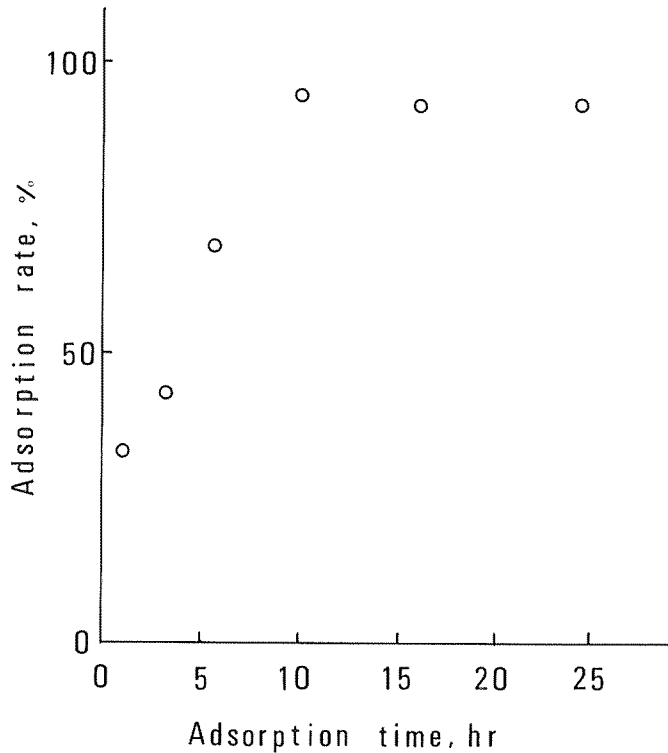


Fig. 2. Effect of adsorption time.

A Hitachi 180-70 Atomic Absorption Spectrophotometer was used.

Procedure

Polypropylene test tubes (6 ml) were cleaned by soaking in 1 : 1 HCl at least overnight. The Au standard, KSCN, HCl, some resin beads and distilled water were put into the test tubes (final volume 3 ml). The test tubes were rotated at 40 rpm. After rotating for 14 h, the beads were transferred to other test tubes. H_2NCSNH_2 in HCl (2.5 ml) was added and the test tubes were rotated at 40 rpm. After rotating for 1 h, the amount of Au desorbed into H_2NCSNH_2 -HCl solution was determined by FAAS.

Results and discussion

Analytical conditions

The effect of the HCl and KSCN concentrations, adsorption time and the number of beads were studied for a solution containing $100 \mu\text{g Au/l}$. Constant values were obtained in the concentrations of 0.5-3.5 M HCl and 0.05-0.2% KSCN. The adsorption time more than 10 h (Fig. 2) and the resin beads more than 5 beads gave a adsorption ratio more than 90%.

The effect of the HCl and H_2NCSNH_2 concentrations on Au desorption was also examined using the same method as in the adsorption study. Constant values were obtained at the concentration of 0.05-0.5 M HCl and 0.05-0.4 M H_2NCSNH_2 . The

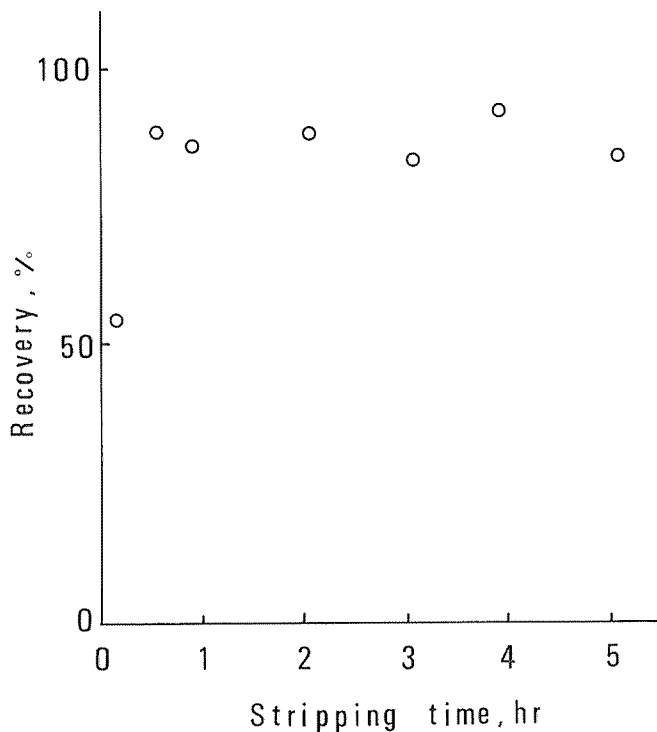


Fig. 3. Effect of stripping time.

Table 1. The overall recovery and the precision

Recovery (%)	Precision
2. 89.9	84.5 ± 3.5 (%) ($\mu = 0.95$)
3. 79.3	
4. 85.2	
5. 87.8	
6. 86.9	
7. 80.2	
8. 82.3	

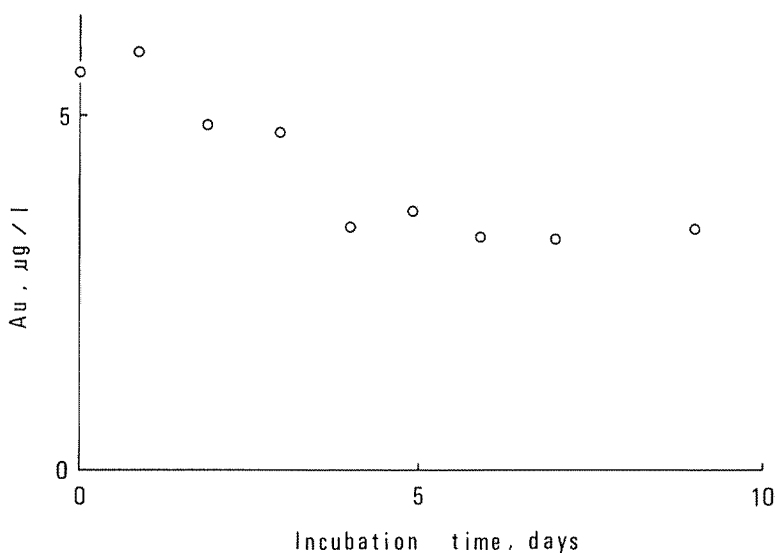


Fig. 4. Uptake of Au by phytoplankton.

desorption time more than 0.5 h gave a constant value (Fig. 3).

Recommended procedure

Add 2.5 ml of a sample solution, 0.3 ml of conc. HCl, 0.1 ml of KSCN and 6 resin beads to the test tube and rotate the tube for more than 12 h. Transfer the beads to an other tube and add 2.5 ml of 0.1 M H_2NCSNH_2 in 0.1 M HCl. Rotate the tube for more than 2 h. Determine the Au concentration by FAAS.

The overall recovery of this method and the precision are shown in Table 1.

As an example sample, this method was used to measure the uptake rate of Au by marine phytoplankton (Fig. 4).

Acknowledgement

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References

- Bruland, K.W., Coale, K.H. and Mart, L. (1985). Analysis of seawater for dissolved cadmium, copper and lead : an intercomparison of voltammetric and atomic absorption method. *Mar. Chem.*, **17**, 285-300.
- Heggie, D.T. (1982). Copper in surface waters of the Bering Sea. *Geochim. Cosmochim. Acta*, **46**, 1301-1306.
- Koide, M., Lee, D.S. and Stallard, M.O. (1984). Concentration and separation of trace metals from seawater using a single anion exchange bead. *Anal. Chem.*, **56**, 1956-1959.
- Van den Berg, C.M.G. (1984). Determination of copper in sea water by cathodic stripping voltammetry of complexes with catechol. *Mar. Chem.*, **164**, 195-207.