Instrumental Achievements

Porous Membrane Phase Separator in Solvent Extraction

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Recently, a solvent extraction-flow injection analysis(FIA)¹⁻³ has been developed which is a useful technique for the pre-concentration of metal ions prior to the determination of metal ions contained in a small volume sample solution by atomic absorption spectrophotometry.

A porous membrane separator is used to develop the solvent extraction-FIA. Consequently, the solvent extraction-FIA is very effective for the determination of trace amounts of metal ions, even those which are contained in a large amount of sample solution.

In this work, the apparatus with a phase separator, shown in Fig. 1, was developed. It was applied to the solvent extraction separation and determination of 10^{-7} mol dm⁻³ level copper(II) ion with 5-octyloxymethyl-8-quinolinol. This liquid is characterized by high hydrophobicity and by high selectivity toward copper(II)⁴ and molybdenum(VI)⁵ in atomic absorption

spectrophotometry.

The apparatus comprises a flat-bottomed reaction flask (1000 cm³), a porous membrane phase separator (Kokubo Seiki, K. K., Fig. 2) (diameter, 10 mm; length, 50 mm), a manometer, and a magnetic stirrer. Nitrogen gas was passed through the flask to separate the organic phase from the aqueous phase by using the porous membrane phase separator while stirring. The pressure of nitrogen gas was adjusted at 30 mmHg. The organic phase comes out through a teflon tube (diameter, 2 mm). The organic phase, once separated, was put into a reservoir, then introduced to the atomizer of the atomic absorption spectrophotometer. When the phase separator was put completely in the solution, the organic solvent came out together with the aqueous solution. Therefore, three-fourths of it, as shown in Fig. 1, was put in the solution. The separator, which was dried at 60°C for 4 h before use, was immersed in a DIBK solution of 5-octyloxymethyl-8-quinolinol.

The effects of the stirring time and of the concentration of 5-octyloxymethyl-8-quinolinol on the extraction of 6.00×10^{-6} mol dm⁻³ copper(II) with 5.00×10^{-3} mol dm⁻³ 5-octyloxymethyl-8-quinolinol at pH 3.0 were investigated for MIBK and DIBK. In the case of MIBK, the difference between the amounts of copper(II) ion found and calculated increased with the increment of the volume ratio of the aqueous phase to MIBK. This should be ascribed to the large solubility of MIBK in water. On the other hand, in the case of DIBK, reasonable results were obtained even for the volume ratio of aqueous phase to DIBK of 60:1. The extraction of a 10^{-7} mol dm⁻³ level copper(II) with 1.00×10^{-2} mol dm⁻³ 5-octyloxymethyl-8-quinolinol reached equilibrium by being stirred for 20 min at pH 3.0.

Figure 3 represents the effect of pH on the extractability of copper(II) with 5-octyloxymethyl-8-quinolinol into MIBK or DIBK. The complete extraction was achieved at pH 1.9 and 2.1 for MIBK and DIBK, respectively.

The effect of diverse ions on the extractive-determination of 5.54×10^{-7} mol dm⁻³ copper(II) with 1.00×10^{-2} mol dm⁻³ 5-octyloxymethyl-8-quinolinol was studied at pH 2.8 for the volume ratio of aqueous phase to DIBK of 60:1. At concentrations up to

Fig. 1 Construction of the apparatus for the separation of metal ion.

Fig. 2 Structure of porous membrane phase separator.

Fig. 3 Effect of pH on the extraction of copper(II) with 5-octyloxymethyl-8-quinolinol (HO_8Q). $[\text{Cu(II)}]$, $3.69 \times 10^{-5} \text{ mol dm}^{-3}$; $[\text{HO}_8\text{Q}]$, $1.00 \times 10^{-2} \text{ mol dm}^{-3}$. Organic solvent, (A) isobutyl methyl ketone, (B) diisobutyl ketone.

$1.00 \times 10^{-3} \text{ mol dm}^{-3}$ each, metal ions including thallium(I), cobalt(II), nickel(II), zinc(II), lead(II), cadmium(II) and chromium(III) did not interfere. In the presence of $1.00 \times 10^{-3} \text{ mol dm}^{-3}$ of iron(III), molybdenum(VI), tin(IV), tungsten(VI), zirconium(IV),

gallium(III) or vanadium(V), the determination of copper(II) was seriously interfered with.

The present apparatus will be applicable to the selective separation and determination of trace amounts of metal ions with highly selective extractant by utilizing the extraction and the subsequent back-extraction procedures.

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