4–6 Preparation of a Micro-Sized Solid-Phase Extraction Cartridge

— Separation Cartridge Designed for Trace Analysis of Difficult-to-Measure Nuclides —

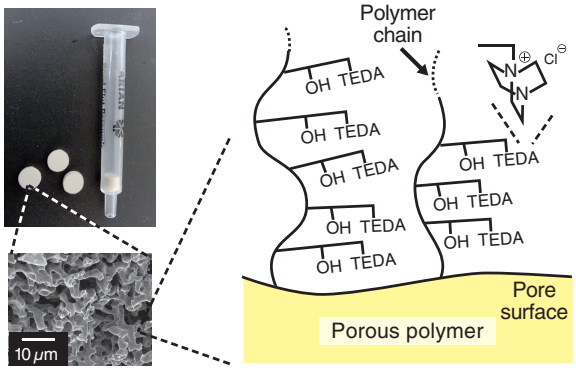

Fig.4-14 Schematic interfacial structure of a micro-volume cartridge (TEDA cartridge)
A disk-shaped highly permeable microporous polymer is packed inside the cartridge. Anion-exchange-group (TEDA)-bound polymer chains, which are closely attached to the polymer surface, enable highly efficient separation.

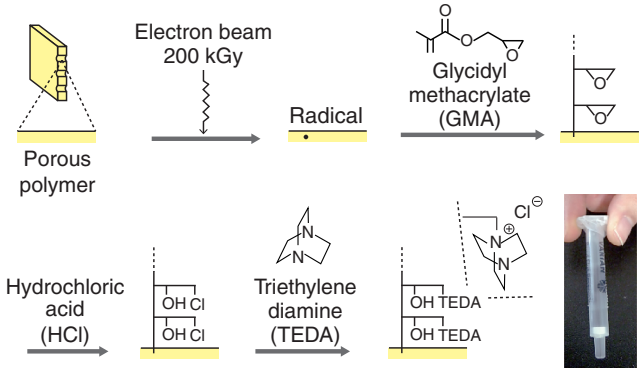

Fig.4-15 Preparation of the TEDA cartridge by electron-beam-induced graft polymerization
Graft polymerization is induced by electron-beam irradiation, producing polymer chains that extend from the pore surface of the polymer. Subsequently, ion-exchange groups are introduced to the polymer chains via reaction with HCl.

Table 4-1 Comparison of separation performances between the TEDA cartridge and conventional adsorbent
The TEDA cartridge can maintain separation performances that are comparable with or higher than those of the commercially available anion-exchange resin-packed column when solutions are fed up to 10 times faster than a typical flow rate for the use of the anion-exchange resin.

	Flow rate (mℓ/min)	Total feeding vol. (mℓ)	^{237}Np recovery	Contamination rate	
				Am	U
TEDA cartridge	1.5	2.5	90%	<0.001%	<0.1%
Commercially available anion-exchange resin ※1	0.15	41.3	78%	<0.001%	2.3%

^{237}Np: neptunium-237
Am: americium
U: uranium

※1 BIO-RAD AG® 1-X8

Radionuclides are commonly determined with radiometry and mass spectrometry. For γ-emitting nuclides, the activities can be measured without any pretreatments owing to strong penetration ability. In contrast, the measurements of α and β-emitting nuclides are associated with pretreatments comprising mostly chemical separations to remove concomitants that may cause interference of the energy spectra. Mass spectrometry, which identifies the mass number of an analyte rather than its radiation energy, also requires separation of isobars and molecular ions with the same mass number. Thus, simplifying such cumbersome and time-consuming separation procedures can be an effective approach to reducing operation time and workload.

Our study has focused upon facilitating the separation procedures by preparing a micro-volume solid-phase extraction cartridge that achieves rapid separation. A disk-shaped, highly-permeable microporous polymer (average pore diameter: 1.0 μm) with a diameter and thickness of 5.9 and 3.0 mm, respectively, is packed inside the cartridge. Triethylenediamine (TEDA), which has two anion-exchange groups per molecule, is uniformly present on the pore-surface of the polymer, forming a high-density ion-exchange-interfacial phase (Fig.4-14). This ideal structure for rapid separation can be realized by the electron-beam-induced graft-polymerization technique (Fig.4-15).

The separation performances of the prepared (TEDA) cartridge were evaluated using a spent nuclear-fuel sample. ^{237}Np, one of the difficult-to-measure long-lived nuclides generated through nuclear reactions, was selected as an analyte. The separation performances of the TEDA cartridge were compared with a conventional chromatography column with a commercially available anion-exchange resin (Table 4-1). The TEDA cartridge could maintain comparable or higher separation performances than those of the conventional column when solutions were fed up to 10 times faster than a typical flow rate for the use of the anion-exchange resin, thus demonstrating that the TEDA cartridge significantly reduced the separation time.

The technique employed for the preparation of the TEDA cartridge is applicable to various molecules that have specific-adsorption ability. Optimal combinations of a molecule to be bound and a base polymer for grafting will provide the capability to meet a wide range of analytical needs.

Reference

Asai, S. et al., Preparation of Microvolume Anion-Exchange Cartridge for Inductively Coupled Plasma Mass Spectrometry-Based Determination of ^{237}Np Content in Spent Nuclear Fuel, Analytical Chemistry, vol.88, issue 6, 2016, p.3149-3155.