RECOVERY OF ION-EXCHANGE RESINS AND PARTITION CHROMATOGRAPHIC SUPPORTS FROM LARGE SCALE RADIOCHEMICAL SEPARATION

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In order to reduce analytical expenses by the re-use of typical ion-exchange resins and partition chromatographic column supports, frequently used in large scale routine analysis of biological samples in many radiochemical laboratories, the problem of their recovery was studied in column and batch operations. The difficulties encountered with respect to some elements are discussed in details.

Introduction

With the need for multielement analysis in biomedical research and environmental pollution control, NAA has found wide applications. Although the pure instrumental approach using Ge(Li) systems has been extensively explored, several limitations necessitate at least some basic radiochemical separations, e.g. in order to obtain the results with in reasonable time. Especially with respect to lower costs, NaI systems are more attractive since many simple, automated, and semiautomated, radiochemical group separation systems using ion-exchange and partition chromatography highly suitable for routine analysis of appreciably large number of samples are available. 1–5,7–12

In routine analysis, however, when a large number of samples are to be analysed, the required supply of column material is considerably high, and, being fairly expensive, influences the cost of analysis to quite an extent.

Although it is of general interest because of the similarity of all these methods, apparently little or no effort has been made for regenerating column materials for repeated use, e.g., even if elution developments are utilized in the separation method itself, low (but still reproducible) recovery values in the range of typically 60–90% must frequently be accepted for many medium- and long half-life radionuclides in order to save time and to avoid more complex working technique. However, diffi-

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cultivates appear inevitably with the repeated use of the system due to the risk of cross contamination from preceding sample processing. The requirement for re-use in serial analysis of similar samples apparently affords elution recoveries well above 90%, and critical considerations are some times necessary even in such cases.

The recovery of larger amounts of used column material, however, may often be achieved by cheap but highly effective regenerating agents, either in a one step operation or in succession, thereby using the contact time needed to attain satisfactory results.

This situation led us in undertaking a set of experiments to recover ion-exchange resins from the elements Ag, As, Br, Cd, Co, Cr, Cu, Fe, Hg, Mo, Sb, Se, Sn, W, Zn by column and batch experiments. The results of these experiments were then applied to recover the column material used in SAMSALH’s automatic ion-exchanger machine (SA-7100). In addition, similar studies were carried out with partition chromatographic column materials to remove the rare earth elements and Sc.

Experimental

Column experiments

New ion exchangers and freshly prepared HDEHP [di-(2-ethylhexyl) orthophosphoric acid] treated Kiselguhr were used in these experiments. Radionuclides of Mo$^{6+}$, W$^{6+}$ as peroxyacids in sulphate medium, Cd$^{2+}$, Ag$^+$ as anionic bromide complexes, Fe$^{3+}$, Zn$^{2+}$, Hg$^{2+}$, Sn$^{4+}$, Sb$^{5+}$ as chloride complexes, Se$^{6+}$, As$^{5+}$ as mixed halogen complexes were adsorbed on anion-exchange resins. In an analogous way Sc$^{3+}$, La$^{3+}$, Ce$^{3+}$ were separated on partition chromatographic columns from a solution of about pH 4. Cu$^{2+}$, Co$^{2+}$, Cr$^{3+}$ were chelated onto a cation exchanger from a solution of about pH 4. A maximum of five identical columns were coupled in series as shown in Tables 1 and 2, and elution was carried out using a peristaltic pump.

Batch experiments

Materials from old columns applied in the separation mode, which were also regenerated as shown in the respective Tables, were used. In case of HDEHP treated Kiselguhr about 30 g were recovered from old columns used to adsorb Sc$^{3+}$, La$^{3+}$, Ce$^{3+}$ in the separation scheme. The lumps were ground to a fine powder and then treated 2 or 3 times with di-isopropylether as a displacing agent. The material was stirred rigourously and kept tightly closed to avoid the vaporization of ether. After filtering off the ether the substance was recovered and regenerated in two batches of 15 g each by treatment with 1.5 g of fresh HDEHP dissolved in 50 ml of di-