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Quantification of ethylene diamine tetra acetic acid in an incinerated ion-exchange resin matrix by solid-liquid phase extraction and RP-HPLC-UV analysis

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Ethylene diamine tetra acetic acid (EDTA) is found in decontamination agents used throughout the nuclear industry, therefore, is often found in repository consigned wastes. The Low-Level Waste Repository (LLWR) is the UK's Centre for the disposal of low-level radioactive waste. LLWR maintain strict limits of acceptance on EDTA because, when present in the waste, the ligand potentially solubilizes otherwise surface-bound radionuclides, making them more susceptible to groundwater transportation into the wider geo/biosphere. A significant quantity of EDTA and radioactively-contaminated ion-exchange resin sourced from decontamination operations during nuclear submarine maintenance is in radioactive decay-storage in UK Naval dockyards. Before the material can be accepted for disposal at LLWR, it must be thermally conditioned to remove EDTA; and then analyzed to confirm EDTA destruction. A simple method has been developed for the extraction and quantification of EDTA from an incinerated ion-exchange resin matrix using reversed-phase ion-pair high-performance liquid chromatography (HPLC) with ultraviolet detection. EDTA is extracted directly into an aqueous Fe (III) solution to undergo complexation, separated on a monolithic silica column (Chromolith® HighResolution, Merck) and detected at 258 nm. The linearity of the response is high (R2; 0.9999) and the limit of detection/quantification for the method has been determined to be 0.23/0.62 mg/kg, respectively. From the standard addition method on four samples of incinerated resin containing 6% EDTA prior to treatment, high recoveries were obtained (mean value; 78.3±3.34 %), with reasonably high intra- and inter-day repeatability (RSD; 1.42–12.4%). Absorption peaks at similar retention times were observed and, as they do not occur for the resin incinerated without EDTA, are attributed to ferric complexes of EDTA thermal degradation products. Interfering peaks were resolved by applying a least squares fit to the data.

Table

Added Standard (mg/kg)	Recovery (%)	Precision (%RSD)	
		Intra-day	Inter-day
1000	81.5 ± 1.64	1.56	2.01
100	79.8 ± 3.07	2.56	3.85
10	78.0 ± 1.11	1.51	1.42
1	73.8 ± 9.14	9.48	12.4
0	0	0	0

Table: EDTA recoveries from the incinerated ion-exchange resin and the repeatability of the procedure - calculated from four trial runs of the standard addition method over two separate days

Recent Publications

1. Wang G and Tomasella FP (2016) Ion-pairing HPLC methods to determine EDTA and DTPA in small molecule and biological pharmaceutical formulations. Journal of Pharmaceutical Analysis 6(3):150-156.
2. Nowack B, Kari F G, Hilger S U and Sigg L (1996) Determination of dissolved and adsorbed EDTA species in water and sediments by HPLC. Analytical Chemistry 68(3):561-6.
3. Kemmei T, Kodama S, Yamamoto A, Inoue Y and Hayakawa K (2013) Determination of ethylene diamine tetra acetic acid in foods by reversed-phase high-performance liquid chromatography. Food Chemistry 138(2-3):866-869.
4. Kemmei T, Kodama S, Fujishima H, Yamamoto A, Inoue Y and Hayakawa K (2012) Determination of ethylenediaminetetraacetic acid in sea water by solid-phase extraction and high-performance liquid chromatography. Analytica Chimica Acta. 709:54-58.

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Biography

James A O'Hanlon is pursuing his PhD at University of Manchester, School of Chemistry, in the group of Prof Melissa A Denecke. He has an Industrial Cooperative Awards in Science and Technology, studentship specially funded by the EPSRC (Engineering and Physical Sciences Research Council) to promote mutually beneficial research collaboration between academic and partner organizations.

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