

## Technetium Speciation in Cement Waste Forms Determined by X-ray Absorption Fine Structure Spectroscopy

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*EXAFS / XANES / Cement / Technetium / X-ray*

### Abstract

The chemistry of technetium in cement waste forms has been studied with X-ray absorption fine structure (XAFS) spectroscopy. Using the Tc *K*-edge X-ray absorption near-edge structure (XANES) as a probe of the technetium speciation, our results show that partial reduction of the pertechnetate ion, TcO<sub>4</sub><sup>-</sup>, takes place in the presence of the cement additive, blast furnace slag (BFS). The addition of the reducing agents FeS, Na<sub>2</sub>S, and NaH<sub>2</sub>PO<sub>3</sub>, produces more extensive reduction of TcO<sub>4</sub><sup>-</sup>, while the compounds FeO, Fe<sub>2</sub>O<sub>3</sub>, and Mn<sub>2</sub>O<sub>3</sub> are observed to be unreactive. The extended X-ray absorption fine structure (EXAFS) data for the BFS, Na<sub>2</sub>S, and FeS treated cements indicate the presence of Tc clusters possessing first shell S coordination. For the Na<sub>2</sub>S and FeS additives, Tc–Tc interactions are detected in the EXAFS demonstrating an extended structure similar to that of TcS<sub>2</sub>. The EXAFS spectrum of the NaH<sub>2</sub>PO<sub>3</sub> treated cement reveals Tc–O and Tc–Tc interactions that resemble those found in the structure of TcO<sub>2</sub>.

### Introduction

Cement-based waste forms are an integral component of national strategies being developed for the safe, long-term storage of low and intermediate-level radioactive waste [1–6]. Numerous physical properties of the cement such as strength, porosity, corrosion resistance, and leachability must be evaluated to effectively encapsulate the hazardous species for long periods of time and under a wide range of environmental conditions. Of these properties, leachability is perhaps the most critical in regards to the immobilization of radionuclides in these matrices. <sup>99</sup>Tc in particular has received considerable attention due to its abundance in nuclear wastes, relatively long half-life, and the high solubility of its most common, oxidized form, the pertechnetate anion (TcO<sub>4</sub><sup>-</sup>).

Because the chemical form or speciation of a radionuclide directly determines many of its properties, modifying the speciation *in-situ* to control solubility and transport is vital for immobilization. In the case of Tc, a decrease in the leaching rate has been achieved through the use of blast furnace slag (BFS)

additives to the ordinary Portland cement (OPC) formulations [7, 8]. The decreased leaching of Tc is believed to result from the reduction of TcO<sub>4</sub><sup>-</sup> to a less soluble form, i.e. Tc(IV) as in TcO<sub>2</sub>, which is brought about by the presence of reducing agents contained in the BFS such as Fe(II) [7] or sulfur-containing species [8]. This hypothesis is supported by thermodynamic data [1] which show that the redox potential in slag blended cements ( $E_{\text{a}} = -300$  to  $-400$  mV) is significantly lower than in other OPC blends ( $E_{\text{a}} = 100$  mV).

We have used X-ray absorption fine structure (XAFS) spectroscopy to determine the Tc speciation induced by the introduction of various chemical additives to the cement formulations. XAFS spectroscopy [9, 10] is an element specific structural technique that is typically divided into two methods concentrating on different portions of an X-ray absorption spectrum. The X-ray absorption near edge structure (XANES) region lies within  $\sim 20$  eV above and below the main absorption edge and provides information about the electronic structure (density of states, oxidation state) and local geometry for a given absorbing atom. The extended X-ray absorption fine structure (EXAFS) region spans the range  $\sim 20$ –1200 eV past the edge and can be used to determine the identity of near-neighbor atoms ( $Z \pm 2$ –6), their coordination numbers ( $N \pm 20\%$ ), and bond lengths ( $R \pm 0.01$  Å) [9, 10]. XAFS does not require long-range order (i.e., crystalline samples) and can be performed on elements in amorphous solids, surface complexes, and aqueous species. This is a key point for the study of Tc and other elements in cement. Recently, XAFS was utilized to investigate the speciation of Tc [11] and Cr [12] in cement-based waste forms. In the present study, the XANES region is used as a sensitive measure of the Tc oxidation state, thereby making it possible to quantify the extent of reduction that takes place in the presence of various additives (BFS, FeS, or Na<sub>2</sub>S) and under different curing conditions. In addition, the Tc EXAFS is used to identify the local structure of several reduced Tc species in the cement waste forms.

# Analytical Aspects Of Technetium Speciation In Spinach Plants

**L Cohen**



## **Analytical Aspects Of Technetium Speciation In Spinach Plants:**

**Analytical Aspects of Technetium Speciation in Spinach Plants** Arend Victor Harms,1998 Bibliography of Agriculture ,1999 **Technetium in the Environment** G. Desmet,C. Myttenaere,2012-12-06 Since the discovery of technetium in 1937 by Perrier and Segre technetium 99 and technetium 99m have become most familiar in the specialised literature the behaviour of the former being of concern regarding its health impact the latter having been used intensively in medical tests for many years The knowledge of the characteristics and behaviour of this element in nature is currently increasing exponentially It has become clear however that there is still a serious shortage of information needed to assess properly its burden and impact on man and his environment Technetium may indeed be released from nuclear installations or as a waste product from medical applications It is generally considered to be highly mobile as the anion  $TcO_4^-$  but it is also suspected to be readily reduced in soils and metabolised in living beings into lower oxidation states The seminar was planned to analyse thoroughly the experimental results obtained up to now and to make recommendations of specific issues requiring further research This book presents the papers of the different sessions in the order of presentation and the conclusions drawn by three discussion groups which dealt separately with problems of sources and distribution of man made technetium and its radiological consequences the chemical behaviour of technetium in different ecosystems biochemical reactions and binding with macromolecules in various life systems vi Preface The editors together with the Programme Committee of the Seminar wish to express their gratitude to the chairmen of the different sessions for their summary of the group discussions

*Trace Elements in Soils and Plants* Alina Kabata-Pendias,2010-10-18 Still the Gold Standard Resource on Trace Elements and Metals in Soils This highly anticipated fourth edition of the bestselling *Trace Elements in Soils and Plants* reflects the explosion of research during the past decade regarding the presence and actions of trace elements in the soil plant environment The book provides information on the biogeochemistry of trace elements *INIS Atomindex* ,1988 Speciation Analysis of Arsenic, Chromium and Selenium in Aquatic Media International Atomic Energy Agency,2007 This publication presents the results of a project analysing the speciation of As 3 As 5 Cr 3 Cr 6 and Se 4 Se 6 in water samples Total element concentration does generally not provide adequate information to understand the effects of trace and heavy metals observed in the environment and in living systems The toxicity bioavailability physiological and metabolic processes and the mobility and fate of elements are to a large extent dependent on the specific chemical form of an element Speciation analysis has yet to be developed to its full potential for biochemical clinical and environmental investigations and still more work is needed in the near future However nuclear techniques particularly the use of radioisotopes can help in method development optimization and stability tests of chemical species *Brinkman's cumulatieve catalogus van boeken* ,1999 Voorts een alfabetische lijst van Nederlandsche boeken in Belgi uitgegeven Brinkman's Cumulatieve catalogus van boeken de in Nederland en vlaanderen zijn uitgegeven of herdrukte ,1998 **Selected Water Resources Abstracts** ,1989 **Selected Water Resources**

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