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Addendum

Addendum to "A modified acid digestion procedure for extraction of tungsten from soil" by A.J. Bednar, W.T. Jones, M.A. Chappell, D.R. Johnson, D.B. Ringelberg [Talanta 80 (3) (2010) 1257–1263]

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Analysis of tungsten has been an issue that has received significant attention recently. The work by Bednar et al. [1] details a procedure for the extraction of tungsten from soil matrices prior to analysis by Inductively Coupled Plasma techniques. The method is modified from a standard soil acid digestion procedure, US EPA Method 3050B [2], which uses nitric and hydrochloric acids and hydrogen peroxide to extract metals from soil matrices. However, method 3050B is not a total dissolution procedure, but rather a strong acid extraction for most metals. Tungsten recovery from standard reference material soils and field soil matrix spike samples containing tungstate and polytungstates using the standard US EPA digestion method 3050B for acid soluble tungsten was low, generally ranging from 25 to 56%. The phosphoric acid modified method reported in Bednar et al. [1], increased acid soluble tungsten recovery to the 86–98% range.

Acid soluble tungsten recovery from the standard reference materials was approximately 100% (compared to solid phase analysis methods, such as Neutron Activation) when a hydrofluoric acid based total dissolution procedure was used, indicating that certain phases present in the reference materials were not susceptible to simple acid extraction, including phosphoric acid. Geochemical phases not extracted using acid techniques (except hydrofluoric acid) include, but are not limited to, silicate minerals [1,2]. Tungsten may also exist in acid-insoluble phases, such as tungsten trioxide (WO_3) [3]. For such acid-insoluble phases, alkaline procedures have been developed [3,4].

Tungsten tends to exist as iron, manganese, or calcium tungstates in natural deposits, although anthropogenic sources also include metallic tungsten, which may form tungsten oxides upon subsequent oxidation [5]. The work of Griggs et al. [6] confirms that WO_3 is insoluble in both common and modified acid digestion techniques, and thus an alkaline alternative is presented. A comparison between acid and alkaline digestions performed with WO_3 spiked soils resulted in 24% recovery using the phosphoric–nitric digestion procedure, whereas 79% recovery was obtained for the alkaline digestion. High tungsten recoveries can be obtained from metallic tungsten and tungstates in soil matrices using phosphoric–nitric digestion procedures [1,7], however, if tungsten trioxide or other acid-insoluble compounds are present, low recoveries will result. Therefore, the conclusion statement in Bednar et al. [1] is clarified by stating the phosphoric–nitric acid digestion procedure described provides quantitative recovery of 'acid extractable' tungsten from soil matrices.

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The use of trade, product, or firm names in this report is for descriptive purposes only and does not imply endorsement by the U.S. Government. The tests described and the resulting data presented herein, unless otherwise noted, were obtained from research conducted under the Environmental Quality Technology Program of the United States Army Corps of Engineers by the USAERDC. Permission was granted by the Chief of Engineers to publish this information. The findings of this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

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