

## Spectrophotometric Determination Of Uranium With Arsenazo

A spectrophotometric method for determining uranium in nuclear waste was developed using 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (bromo-PADAP). By extracting the color into Aliquat-336 it was possible to accurately measure U at the 1  $\mu\text{g}$  level. No significant interferences were observed from 34 interfering ions when a tri-n-octylphosphine oxide (TOPO) pre-extraction of U was used. The effect of pH, color development time, bromo-PADAP concentration, and Aliquat-336 concentration were determined. The method was applied to the analysis of alkaline solutions, filtered solids, sludges, and salt cake nuclear waste forms. The standard deviation for the method with a TOPO pre-extraction was  $\pm 3.7$  percent.

PADAP is a very sensitive reagent for the determination of uranium. The PADAP method is relatively unaffected by the presence of associated ions, particularly if a compensating blank is applied. Only Cr(3+), As(5+), PO<sub>4</sub>(3-), and V(5+) have been found to give any serious interference, and the levels of these ions in Witwatersrand ores are usually well below those likely to cause interference. The presence of aluminium extracted from the ores will increase the level at which As(5+) and PO<sub>4</sub>(3-) interfere. A comparison of the PADAP method with the thiocyanate method shows that the former has advantages in sensitivity, tolerance for interfering ions, and, possibly, precision. If the PADAP procedure is combined with an extraction step, the tolerance of the method for interferences is increased to an extent where the ions introduced by complete dissolution of a 2 g sample of a silicate ore do not interfere. By the use of this technique it has been shown that the insoluble uranium in Witwatersrand silicate ores varies from 4 to 10 ppm of U<sub>3</sub>O<sub>8</sub>.

The thiocyanate-TBP method has been used daily at the National Institute for Metallurgy for approximately nine years for the determination of small amounts of uranium in Witwatersrand conglomerate and other siliceous ores. It replaced a spectrophotometric method that required the separation of uranium from most other elements in the sample by extraction with ether. The method involves the formation of uranyl thiocyanate complex of uranium in the presence of EDTA and sodium formate, the extraction of the complex into tri-n-butyl phosphate (TBP), and the measurement of the transmittance of the solvent phase. The procedure is applicable to ore samples, filter cakes and aqueous solutions of low uranium content. The thiocyanate-TBP method has several advantages over the 'ether' method.

An ultraviolet spectrophotometric method for the determination of uranium has been developed which is based on the ultraviolet absorption of the complex of uranium(VI) with tris-(2-ethylhexyl)phosphine oxide. The complex is formed by extracting uranium(VI) from an aqueous 6M sodium nitrate solution in the pH range 2.5-3.0 into 0.1M solution of tris-(2-ethylhexyl)phosphine oxide (TEHPO) in an inert diluent, cyclohexane.

A method for the direct determination of uranium in a cyclohexane solution of tri-n-octylphosphine oxide (TOPO) is presented. The adduct, UO<sub>2</sub>/Cl<sub>2</sub> x 2TOPO, that is formed when uranium(VI) is extracted from hydrochloric acid solutions by tri-n-octylphosphine oxide absorbs light in the ultraviolet region. This absorbance is measured at 230 m $\mu$  vs. a TOPO-cyclohexane solution that was contacted with hydrochloric acid of the same concentration as that in the test aliquot. The molar absorbance index is 5500. The method is not selective; of the elements that are extracted by TOPO from hydrochloric acid, iron(III), zirconium, molybdenum, tin and thorium, only thorium can be tolerated. (auth).

The Spectrophotometric Determination of Uranium in Mixtures of Uranium and Tungsten  
The Spectrophotometric Determination of Uranium by Means of the Azide Ion  
Ultraviolet Spectrophotometric Determination of Uranium  
Separation of Uranium from Bismuth Using Tris-(2-ethylhexyl)phosphine Oxide

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