

Determination of Thorium in High Purity Aluminum by ICP-MS

Abstract:

A combined matrix removal by on-line solid phase extraction and ICP-MS method has been developed for the determination of thorium in high purity aluminum. The sample solution prepared as 3M HNO₃ solution was loaded onto a column packed with TEVA Resin (Eichrom Technologies, Inc, Darien, IL, USA.) Thorium is retained on the column and then eluted by 1M HCl solution. The effluent was directly introduced into the nebulizer of an ICP-MS instrument and thorium was measured continuously at a mass number of 232. Thorium at ppb or sub ppb levels in high purity aluminum reference materials provided by the Japan Society of Analytical Chemistry was determined accurately and precisely.

Introduction.

Thorium in nature consists of almost 100% ²³²Th, which is a radioisotope with a very long half-life (1.4 x 10¹⁰ years.) The alpha particles emitted by even a very small amount of thorium in a large scale integrated circuit (LSI) materials induces soft errors in the CPU of a computer and the strict control of its concentration in LSI materials at ppb levels (or less) is important. Inductively coupled plasma mass spectrometry (ICP-MS) is a suitable analytical technique for measuring this level of thorium. Because this technique is susceptible to matrix interferences, it is absolutely necessary to separate the thorium analyte away from other matrix components prior to injection into the plasma to ensure accurate measurement of thorium values.

TEVA Resin from Eichrom Technologies is a specialty chromatographic material that has a very high affinity for tetravalent thorium from nitric acid solutions and a low affinity for most mono-, di- and tri-valent metal ions, including aluminum (see figure 2&3.) The retention of Th is quite low from HCl solutions and could be used to elute thorium sorbed on to the column. This resin was used to remove matrix components of the samples for thorium analysis prior to injection in the ICP-MS instrument.

Experimental:

Aluminum samples (20 – 200 mg) were dissolved in 3 mL each of HNO₃ and HCl with heating followed by evaporation to near dryness. The residue was treated with 3 mL each of HNO₃ and HCl, followed by 3 mL of HNO₃ and finally, the residue was taken up in 5 mL of 3M HNO₃.

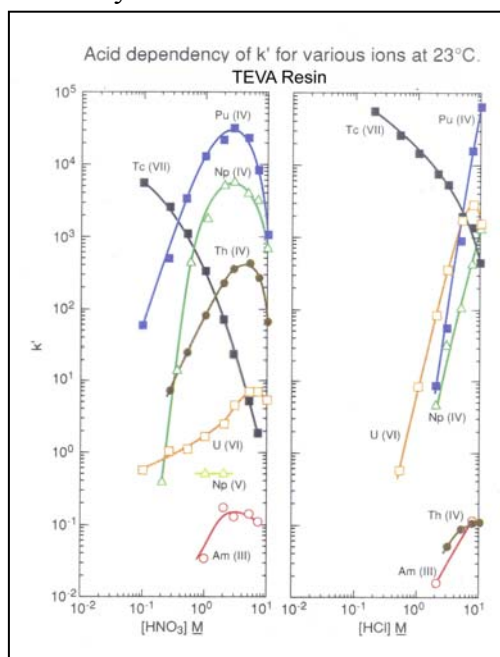


Figure 1

A flow injection system was used to accomplish the matrix elimination on-line. The separation column was prepared by packing 3 g of TEVA Resin in a 4.6 x 50 mm PEEK column. The column was conditioned with 3M HNO₃ for 1 minute at a flow rate of 3 mL/min. Samples prepared as above were passed through the column at 3 mL/min flow rate to sorb thorium on the TEVA Resin column. Following a 2 minute rinse with 3M HNO₃ to remove aluminum, the thorium was eluted with 1M HCl in a reverse direction flow. The column effluent was introduced directly into the nebulizer of the ICP-MS system (Seiko, SPQ9000, Tokyo, Japan.) The operating conditions for the ICP-MS are shown in Table 1.

Plasma Conditions	
Rf power	1kw
Nebulizer gas flow	1.0 L/min
Plasma gas flow	16.0 L/min
Auxiliary gas flow	1.0 L/min
Sampling conditions	
Sampling depth	10 mm
Sampling cone	Copper, 0.80 mm orifice
Skimmer cone	Copper, 0.40 mm orifice

Table 1. ICP-MS Operating Conditions

Results

Sample	Size (g)	Th found (ng/g)	Certified value (ng/g)
JAC0021	0.0268	10.7	
	0.0260	11.3	
	0.0279	10.7	
	Mean:	10.9 ± 0.35	
JAC0022	0.0808	1.4	
	0.0846	1.4	
	0.0811	1.7	
	Mean:	1.5 ± 0.2	
JAC0023	0.3120	0.099	
	0.2726	0.073	
	0.2797	0.089	
	0.2608	0.084	
	0.3181	0.091	
	0.2762	0.080	
	Mean:	0.086 ± 0.009	
		0.086 ± 0.037	

Table 2 Determination of thorium in high purity aluminum reference materials.

Various high purity aluminum reference materials supplied by the Japan Society for Analytical Chemistry were analyzed for thorium content using the method described above. These results are summarized in table 2. Each analytical value is in good agreement with the corresponding certified value and precision is excellent.

The sample throughput is **five samples per hour** when a 10 mL sample is used and the **detection limit of 8 pg** was achieved (3σ of background noise.)

Reference:

Seki, T. & Oguma, K., J. Flow Injection Anal. Vol. 18, No. 2, (2001) 140-143.

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