



Determination of Impurities in High Purity $ZrCl_4$ Material by ICP-MS after Separation of the Matrix using D2EHPA and ZrO_2 Nanostructure Product

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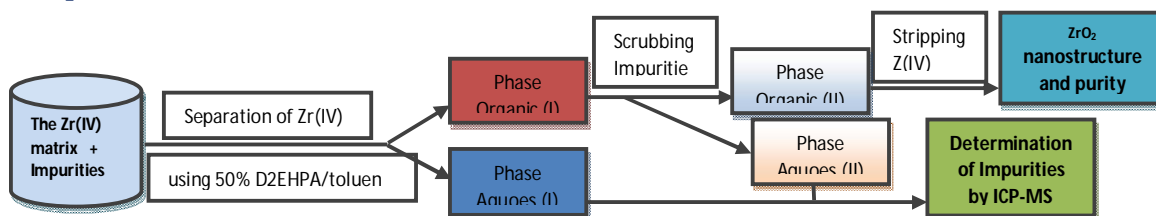
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ABSTRACT

ICP-MS using matching matrix and internal standard is believed direct determination some impurities of high content in high purity $ZrCl_4$. However, trace impurities need to be separated from the Zr matrix to eliminate the interference of the matrix and determination of them by ICP-MS using the internal standard (as In, Bi). The study on capability extraction of Zr(IV) by di-2-ethylhexyl phosphoric acid (D2EHPA) were examined by infrared spectrum (IR) of $ZrO(NO_3)_2$ salt, D2EHPA-toluene solvent and Zr-D2EHPA-toluene complex. Impurities in $ZrCl_4$ were also determinate when using internal standard indium (In) after separation of them from the matrix Zr by extracting in 50% of dissolved D2EHPA in toluene. Investigation of separation of impurities from the matrix Zr showed that with using 50% D2EHPA/toluene solvent, after one cycle extraction using 3M HNO_3 and 1-2 cycles stripping Zr and scrubbing impurities by 6M HNO_3 can recovery for 95-100% of almost investigated impurity elements and stripping about 20-26% of Zr(IV) by ICP-MS using internal standard In. Our results indicated that with the mentioned amount of Zr, effect of the matrix Zr on the determination of almost elements by ICP-MS can be negligible. Levels of impurities were relative standard deviations (RSD) less than 8.4% and recovered (Rev) of 91.7-105.5%, so determination of impurities was high reliability and accuracy. After extraction of the Zr matrix in 3M HNO_3 and back-extraction by 1.5M H_2SO_4 , stripping about 98,7% of the matrix Zr come back in aqueous phase and to get new ZrO_2 product. The energy dispersive X-ray (EDX) of new ZrO_2 product showed that it is purity. The X-ray diffraction (XRD) and transmission electron microscopy (TEM) showed that the crystal structure and morphology of new ZrO_2 product are spherical and nanostructure, which can be applied on the treatment of metal ions in wastewater sources and anti-corrosion steel.

Graphical Abstract



Highlights:

- Separation impurities from the Zr(IV) matrix by solvent extraction using D2EHPA/toluene.
- Determination of impurities after separation by ICP-MS.
- Purity ZrO₂ nanostructure and apply on wastewaters or anti-corrosion steel.

Keywords: Impurities, ZrCl₄, Extraction, D2EHPA, ICP-MS, ZrO₂ Nanostructure.
