

The Extraction of Surface and Bulk Trace Metal Impurities from Typical Fluoropolymers

Dennis Chilcote and Donald C. Grant, CT Associates, Inc.

Samantha Tan, ChemTrace Corporation

Debra Carrieri, FSI International

Abstract

Design specifications for metals contamination from components in chemical handling systems are being established to improve the quality of high-purity semiconductor process chemicals that these systems deliver. The program to develop specifications requires some concomitant methodology to determine whether or not a given component meets the specification, such as the DyconE^{x SM} technology that was developed and patented by FSI International. The objective of this study was to evaluate which acids should be used in this type of test method.

The extraction of trace metal impurities from both perfluoroalkoxy (PFA) and fluorinated ethylene propylene (FEP) bottles was evaluated over a 100-day period using concentrated hydrofluoric, hydrochloric, nitric and sulfuric acids. Each type of bottle was filled with a single ultrapure acid, except for the hydrofluoric acid tests, which were run in triplicate. A sample of acid from each bottle was analyzed at 0.01, 0.54, 3, 18, and 100 days. The analytical techniques used for the analysis of trace metals were inductively coupled plasma/mass spectroscopy (ICP-MS) and graphite furnace atomic absorption spectroscopy (GFAAS). The extraction of trace metals during the first 15 minutes (the 0.01-day sample) of each test was designated as surface extraction, whereas extraction between 0.01 and 100 days was designated as bulk extraction.

Although there is sufficient scatter in the triplicate data to suggest caution in drawing hard conclusions on certain comparison, many general trends were observed. The major leachable trace metals from both types of bottles included Al, Ca, Fe, Mg, K and Na. Surface extraction was comparable to bulk extraction from the FEP bottles for all acids. Except for one hydrofluoric acid replicate, bulk extraction significantly exceeded surface extraction from the PFA bottles for all acids. For both bottle types, sulfuric acid was the least aggressive extractant. There did not appear to be any significant shift in the fingerprint of trace metals between surface extraction and bulk extraction for other types of bottle. Significant extraction of certain trace metals occurred during the time interval between 18 and 100 days. Except for sulfuric acid, the data do not support the conclusion that one acid is superior to all others.

Since samples were taken over time, rates of extraction could be estimated based on a power law model of the extraction of trace metals from a flat surface of bulk fluoropolymer. Rates of extraction for hydrofluoric, nitric and hydrochloric acids are of the same order of magnitude, but are on order on magnitude lower for sulfuric acid. Given the limited data of this study, the similarity in the rate data for hydrofluoric, nitric and hydrochloric acids suggest that any one of these acids could be effectively used to evaluate the extraction of trace metals from fluid handling components. The clear conclusion from this study is that sulfuric acid should not be used as a test acid.