NANOPARTz™

The gold nanoparticle for nanotechnology

ICP-MS Gold Analysis Protocol
1/8/2009

1. Digestion. The sample is heated to 28C until the solution appears clear and then sonicated for > 5 minutes, and vortexed for > 30 minutes at temperature. An initial volume of 0.1mL of each sample is used. One milliliter of nitric acid and three milliliters of hydrochloric acid are added. The samples are then digested for approximately 4-6 hours in a hot block to near boiling. The samples are then cooled and brought up to a final volume of 50mL. An aqua regia microwave digestion is performed as sample preparation step, applying the following procedure: 5 min at 250 W, 5 min at 400 W, 5 min at 650 W and 5 min at 250 W. (update 3-20-12)

2. The samples are analyzed following SW-846, 3rd Edition procedures. Analysis by ICP-MS followed method 6020A and SOP 827 Rev. 7. The relationship between intensity and concentration for each element is established using at least four standards, one of which is a blank solution. A calibration equation relating instrument response to concentration is developed by the instrument software. The equation is a higher order polynomial. This type of equation is used to improve quantitation accuracy at lower concentrations where the relationship between concentration and instrument response is non-linear. During sample analysis concentrations are computed by the software and the results are printed in ug/L. The validity of the calibration equation is tested by analyzing the following solutions: a blank, a low level check solution with concentrations near the reporting limit, an Initial Calibration Verification (ICV) standard from a 2nd source standard solution with concentrations near the middle of the analytical range, a Continuing Calibration Verification (CCV) standard with concentrations near the middle of the analytical range but different than those in the ICV, and a readback of the highest calibration standard. These solutions provide verification that the calibration equations are functioning properly throughout the analytical range of the instrument. During sample analysis dilutions are made for analytes found at concentrations above the highest calibration standard. No results are taken from extrapolations beyond the highest standard.

3. All standards and solutions are NIST traceable and are used within their recommended shelf life.

4. The samples are prepared and analyzed within the established hold times. All in house quality control procedures are followed, as described below.

5. General quality control procedures. A preparation (method) blank and laboratory control sample are digested and analyzed with the samples in this digestion batch. The preparation (method) blank associated with this digestion batch should be below the reporting limit for the requested analyte. The laboratory control sample associated with this digestion batch should within the acceptance limits. This indicates complete digestions according to the method. All initial and continuing calibration blanks associated with this analytical batch should be below the practical quantitation limits for the requested analyte. All initial and continuing calibration verifications associated with this analytical batch should be within the acceptance criteria for the requested analyte. This indicates a valid calibration and stable instrument conditions. The high

standard readbacks associated with Method 6020A should be within acceptance criteria. The interference check samples associated with Method 6020A should be analyzed.

6. The samples will require dilutions to bring gold into the analytical range of the ICP-MS.

7. Three samples for each lot are analyzed and their results averaged.