

X-RAY FLUORESCENCE ANALYSIS OF PALLADIUM IN NON-HOMOGENEOUS ORGANICS AND SLURRIES VIA GEL SUSPENSIONS

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Palladium is a commonly used catalyst for many organic syntheses and the recovery of the spent catalyst is important economically. One way to measure palladium is by using inductively coupled plasma (ICP) atomic emission spectrometry. Volatile organics in the samples are removed by evaporation under nitrogen and the residue is digested in fuming sulfuric acid followed by the addition of nitric acid. The preparation of the samples for ICP analysis has a number of issues: long preparation/analysis time (4-6 hrs), palladium recovery issues, and safety concerns.

Using a commercially available gelling agent (Aquasol-2™), an alternative method was developed where stable gel suspensions are prepared for X-ray fluorescence (XRF) analysis. This technique allows the analysis of non-homogeneous process samples in difficult matrices, where palladium ranges from <10 ppm to percent levels. Relative to the current ICP analysis, the use of gel suspensions coupled with XRF analysis has the advantages of being simpler, faster and safer without sacrificing accuracy and precision. Initially, the technique was developed using wavelength dispersive XRF. Further evaluation was done using energy dispersive XRF equipped with polarizing secondary target technology.