



The Determination of Platinum and Palladium by Inductively Coupled Plasma Optical Emission Spectrophotometry (ICP-OES)

Florin Analytical
Services

February 2017

• 7950 Security Circle • Reno, Nevada 89506 • Telephone: 775-972-7575 • Web: florinanalytical.com • E-mail: fas@florinanalytical.com •

Platinum and Palladium Analysis

This information circular describes the general procedure utilized by Florin Analytical Services (FAS) for assaying platinum (Pt) and palladium (Pd) in ores, concentrates and industrial products.

Fire assaying is the preferred method for the separation of platinum and palladium from gangue minerals and pre-concentration of the precious metals prior to analysis by instrumental techniques.

Assay Procedure

FAS utilizes fire assay lead pre-concentration methods prior to analysis for platinum and palladium by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES).

Fusion Process

A known sample weight (typically 30 grams) of pulverized geological material is thoroughly mixed with flux. The flux consists of a mixture of litharge (PbO), soda ash (Na₂CO₃), borax (Na₂B₄O₇), silica (SiO₂), flour and silver (gold inquart can be an alternate). The mixture of flux and sample is placed into a fire-clay crucible that is then loaded into a furnace set at 1050°C and allowed to fuse for 60 minutes. During the fusion process, the litharge is reduced to metallic lead by the flour and the lead scavenges the precious metals.

Cupellation Process

When the fusion is complete, the crucible is removed from the furnace and the molten fusion product is poured into a conical mold. The lead, with the precious metals collected, drops to the bottom of the mold and is allowed to cool. After cooling, the lead button is removed from the mold and separated from the slag. The lead button is then placed into a pre-heated cupel in a muffle furnace set at 950°C. The cupellation process, where lead is converted to lead oxide and absorbed into the cupel leaving a doré bead of precious metals, takes between 30 and 40 minutes. The cupel is removed from the furnace and allowed to cool to room temperature. Any discolorations or sprouting found on the bead after cooling is noted in the assay worksheets.

Digestion

The doré bead is removed from the cupel and quantitatively digested with nitric acid followed by hydrochloric acid. The resulting solution is analyzed for Pt and Pd by ICP-OES.

Solution Analysis

FAS utilizes a Perkin-Elmer Optima 8300 DV or 2100 DV ICP-OES to analyze for platinum and palladium in solutions. The fire assay pre-concentration procedure followed by ICP-OES is effected by a minimal amount of spectral interferences. By utilizing ICP-OES, these interferences can easily be visualized and eliminated.

Comments

The fire assay lead collection technique utilized by FAS is an excellent method for the quantitative analysis of platinum and palladium. Blanks and commercial standards are run with each job to monitor for the presence of platinum and palladium. Spectral interferences are examined closely on each run and a multi-line spectral analysis are conducted for each element in order that any significant interferences are identified and taken into account.

FAS routinely assays samples for platinum and palladium in duplicate (high grade samples in triplicate) and reports the results of each separately. Slag and cupel material are routinely re-assayed to confirm the quantitative collection of the platinum and palladium values. FAS has spent a considerable amount of resources in developing its ICP-OES technique. FAS has participated in several round-robins with other groups utilizing similar and differing finishing techniques. These comparisons were utilized in the development of the method and are now utilized as part of on-going QC and QA protocols.