

Method Validation for Determining Platinum and Palladium in Catalysts Using Inductively Coupled Plasma Optical Emission Spectrometry

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Abstract : The study presents the analytical capability and validation of a method based on microwave-assisted acid digestion for quantitative determination of platinum and palladium in catalysts using inductively coupled plasma optical emission spectrometry (ICP-OES). In order to validate the method, the main figures of merit such as limit of detection and limit of quantification, precision and accuracy were considered and the measurement uncertainty was estimated based on the bottom-up approach according to the international guidelines of ISO/IEC 17025. Limit of detections, estimated from blank signal using 3 s criterion, were 3.0 mg/kg for Pt and respectively 3.6 mg/kg for Pd, while limits of quantification were 9.0 mg/kg for Pt and respectively 10.8 mg/kg for Pd. Precisions, evaluated as standard deviations of repeatability (n=5 parallel samples), were less than 10% for both precious metals. Accuracies of the method, verified by recovery estimation certified reference material NIST SRM 2557 - pulverized recycled monolith, were 99.4 % for Pt and 101% for Pd. The obtained limit of quantifications and accuracy were satisfactory for the intended purpose. The paper offers all the steps necessary to validate the determination method for Pt and Pd in catalysts using inductively coupled plasma optical emission spectrometry.

Keywords : catalyst analysis, ICP-OES, method validation, platinum, palladium

Conference Title : ICACAS 2019 : International Conference on Analytical Chemistry and Applied Spectroscopy

Conference Location : Venice, Italy

Conference Dates : June 20-21, 2019