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Certification of Pd and Pt single spikes and application to the quantification of Pt and Pd in automotive exhaust emissions

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Numerous epidemiological studies show the effect of increased ambient pollution. Therefore measurement networks for air quality have been installed worldwide and legislation requires the monitoring of air pollution. Besides monitoring it is also important to be able to identify, to quantify and finally to regulate the emission of distinct sources in order to improve the quality of life.

Automotive vehicles are a major source of environmental pollution especially through contaminants such as CO, NOX, SOX and hydrocarbons which derive from petrol combustion, while for example Platinum Group Elements (PGE) can be present from catalytic converters. The release of PGE into the environment, however, may be damaging in terms of public health, ecological and economic interests. In order to reliably assess the risks from PGEs, traceable and thus comparable data on the release rates of PGE from automotive catalysers are needed. As no Certified Reference Materials (CRM) are available for such samples the development of analytical procedures enabling SI-traceable results will be challenging. Therefore reference procedures for Pd and Pt in automotive exhaust emissions based on isotope dilution mass spectrometry (IDMS) have been developed and applied to specifically sampled automotive exhaust emissions.

Due to the commonly known advantages, IDMS often is applied for quantification PGEs, as is the case within this work. The main reasons here are the required accuracy and the low PGE mass fractions in the sample. In order to perform IDMS analysis the analyte element must be available in an isotopically enriched form as so-called spike material or solution thereof, which is mixed with the sample. Unfortunately, no certified PGE spike solutions are available yet.

To fill this gap two single PGE spikes, one 106Pd and one 194Pt spike, have been produced and characterized. The selection of the isotopes, the production of the solutions and the ampoulation will be described in this presentation. Details on the characterization of these spike solutions by reverse IDMS using a primary assay for Pd and Pt will be given. With measurement uncertainties < 0.1 % for the Pd and Pt mass fraction both spike solutions are well suited to become certified reference materials under the ERM® label.

The newly developed IDMS reference procedures consist of a dissolution step by microwave assisted digestion or wet high pressure ashing, followed by a 2-step ion chromatographic Pd- and Pt-matrix separation. The total blank values for this analytical procedure are ≤ 5 pg for Pd and ≤ 75 pg for Pt. First results on filters obtained under harmonized driving cycles (e.g. Artemis, NEDC) show Pd and Pt masses below 1 ng down to 100 pg with some filters showing relatively high values of 4 – 6 ng, which of course depends on the driving cycle.