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EVALUATION OF A MICROWAVE-ASSISTED ACID-DIGESTION AND A DILUTE-AND-SHOOT PROCEDURES FOR OIL ANALYSIS

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Inductively coupled plasma-mass spectrometry (ICP-MS) is a multi-element technique suitable for the analysis of liquid samples, which provides superb selectivity, sensitivity and lower detection limits[1]. ICP-MS is an excellent tool for detailed characterization of the elemental composition of numerous samples, including fuel and lubricating oil samples. The direct introduction of petroleum-derived products for direct analysis using ICP-MS generally requires dilution with an organic solvent. The introduction of an organic solvent into an ICP can dramatically alter the performance in terms of signal intensities or even cause plasma extinction, especially if volatile solvents are used[2]. Some strategies can be adopted for avoiding plasma extinction: reduction of the sample uptake rate, reduction of the nebulizer gas flow rate, increase of the applied power, and addition of oxygen[3]. An ICP-MS equipped with a dynamic reaction cell (DRC) is utilized actively to promote chemical reactions between a reactive gas and interferences (or analyte) to overcome interference processes. The DRC consists of an enclosed cell pressurized with a reactively gas through which the ion beam is passed, for remotion of isobaric interferences. In this work we tested two sample preparation procedures for lubricating and diesel oils: digestion of 100 mg of sample or certified reference materials (CRM) in a pressurized microwave, and subsequent introduction in ICP-MS with a cyclonic spray chamber and Meinhardt concentric nebulizer as sample introduction system and another procedure of direct dilution of 1.0 mg of oil samples or 50 mg of CRM diluted to 10.0 or 20.0 g with kerosene, respectively, and subsequent introduction in ICP-MS using a PFA spray chamber and a micro-concentric nebulizers. Measurements were carried out using an ELAN DRC plus: power 1350 W, plasma gas: 15 L min^{-1}, nebulizer gas: 0.95 L min^{-1}, and DRC nebulizer gas 0.98 L min^{-1} NH_3. Using the cyclonic spray chamber the sample uptake rate was 1.2 mL min^{-1} and with the micro-nebulizer the sample uptake rate was 20 µL min^{-1}. Determinations of Cr, Cu, Fe, Ni, and Zn were performed with DRC on, for overcoming isobaric interferences. Both procedures are feasible but better detection limits were reached using the dilute-and-shoot approach.

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