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Use of Factorial Design for Comparison of Two Liquid Sample Introduction Systems for an Axially Viewed ICP OES

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Axially viewed inductively coupled plasma optical emission spectrometers are known for the improvement in sensitivity when compared to radially viewed systems. However, the system performance could be degraded due to the increment of interferences. It is usually accepted that the minimization of interferences can be obtained when a proper interface system is used (end-on gas or shear gas) and when adequate operating parameters are employed, such as a high applied power and a low nebulization gas flow-rate, along with an increase in the i.d. of the injector. The use of these operating conditions should lead to robust plasma, where the temperature and the electron number density are not significantly modified. In this work, a factorial design is proposed to identify the influence of operating parameters on plasma conditions. An axially viewed ICP OES (Varian) was used in this work. The system was set with a simultaneous CCD solid-state detector and an end-on gas interface was used to minimize interferences. Two different liquid sample introduction systems were used: a V-groove nebulizer with a Sturman-Masters spray chamber and a concentric nebulizer with a cyclonic spray chamber. The robustness (Mg II / Mg I ratio) of the system was calculated by monitoring Mg I 285.208 nm and Mg II 280.264 nm emission lines for solutions containing 2 mg L⁻¹ of Mg in acid and alkaline media. The influence of applied power and carrier gas flow rate was evaluated using a 2² central composite design and using a triplicate of the central point. Applied power was varied from 0.7 to 1.4 kW and nebulizer gas flow-rate was varied from 0.6 to 1.4 L min⁻¹. The use of this factorial design allowed the construction of a response surface. Results showed that robust conditions were obtained at higher applied power and lower carrier gas flow rate when using a concentric nebulizer and a cyclonic spray chamber. On the other hand, when using a V-groove nebulizer and a Sturman-Masters spray chamber, robust conditions were reached at higher flow rates. This is unexpected but may be related to the influence of the sample introduction system on total mass of analyte and solvent transported towards the plasma and the aerosol characteristics. The aerosol drop size changes the plasma location at which the drop vaporization is complete. In both cases, the use of robust conditions was essential for the recovery of the elements in milk samples diluted in alkaline medium.

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