

FACTORIAL DESIGN FOR METHOD DEVELOPMENT AIMING AT THE DETERMINATION OF CD AND PB IN CARROT AS ACIDIC SLURRY BY GF AAS FOR COMPLIANCE WITH INTERNATIONAL FOOD LEGISLATIONS

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Determining inorganic contaminants in vegetables by atomic spectrometry techniques remains as an analytical challenge. Considering Pb determination in vegetables by GF AAS, for instance, a severe sensitivity barrier is found when microwave oven (MW) is employed for sample digestion because of the sample mass restriction, which makes the analysis impracticable when intending compliance with international food regulations. Beyond the mass limitation, such classical approaches for sample digestion (block digester included) mandate employment of large volumes of acids, which are not environmentally friendly and are highly destructive to the spectrometers' parts, like graphite tube in GF AAS, which will then force large digest's dilution, leading to incompatibility with the low levels allowed for such compounds in international food legislations. Furthermore, an analytical method for official control of elements must be as simple as possible, reliable, present high sample throughput and a limit of quantification less than one fifth of the maximum level for the element, according to EC. The objective of this work was to develop a method for cadmium and lead determination in carrot by GF AAS aiming at compliance with international food regulations. Acidic slurry approach was investigated for Cd and Pb determination in dried carrots by GF AAS. A 2² factorial design was employed (for factors sample mass and HNO₃ volume), which provided recoveries of about 80% for both analytes for most of the combinations. The optimum experimental conditions for acidic slurry, compromising Cd and Pb responses following Pareto charts evaluation, were 200 mg of dried carrot plus 1 ml of HNO₃, submitted to 2 hours of ultrasound bath, and sample final volume of 5 ml. Acidic slurries of carrots has showed simplicity and high efficiency, without the need of a complex sample preparation procedure or instrumentation.

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