Título: VALIDATION OF AN ANALYTICAL METHOD FOR THE DETERMINATION OF CADMIUM (Cd) IN FISH BY AAS WITH ELECTROTHERMAL ATOMISATION

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RESUMO

The validation of an analytical method was carried out for the determination of cadmium (Cd) in fish. The method was based on sample digestion in a microwave oven and subsequent reading using an atomic absorption spectrometer with a graphite furnace. The factorial design of experiments was applied to assess method ruggedness using the methodology of Box et al. [Box GEP, Hunter WG, Hunter JS. 1978. Statistics for experiments: an introduction to design, data analysis and model building. New York (NY): Wiley], studying the influence of sample mass, volume and concentration of acid used for sample digestion and the volume of modifier used. To study the possible matrix effect in the determination of Cd, the standard addition method was also performed. The results were treated using the OLS method. For the normality test a homoskedastic distribution was observed for the developed method and the results were adjusted to the statistical model proposed. F-tests and Student’s t-tests indicated that there was no matrix effect on the calibration curve between the concentration range 1.0–10.0mgCd/l. Parameters such as selectivity, precision, decision limit, detection capability and limit of quantification were established by the method of standard addition to blank samples. The limit of quantification was 6.8mgkg–1. Accuracy, which was evaluated by using a certified reference material, was 107.0%. The recovery of the spiked analyte was 93.69% for the concentration of 50mgkg–1. Precision was defined by the coefficient of variation observed (Horrat value), estimated in terms of repeatability and reproducibility, and the values were below the limit, which is 2.0. The validation procedure confirmed the suitability of the method. A method for determination of cadmium using the technique of graphite furnace spectrometry was validated according to the requirements set by the European Union. The validation results indicate that the method is precise and accurate and that the limit of quantification meets the criteria set out in Regulation 2007/333/EC. Therefore, the method is suitable for use on official control, because the quality and compar-ability of analytical results can be ensured.