

Validating the analysis method for Cd, Cr and Pb in ceramics using ICP-AES

Aurélie MOAL

Introduction

Before being allowed to market, manufacturers of materials and articles in contact with foodstuff have to test their products. This typical test determines the release of chrome, lead and cadmium from ceramic ware. Indeed, heavy metals are severely supervised because of their toxicity if they are absorbed in large amounts. This work aimed at validating a method to measure of 3 elements: Cadmium (Cd), Chrome (Cr) and Lead (Pb) by ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry) in aqueous extracts of ceramics (4% acetic acid in water). The European standards NF EN 1388-1 and 1388-2 impose that Cd, Cr and Pb concentrations do not exceed the following values : [Cd]=0.1 mg/L, [Cr]=0.03 mg/L and [Pb]=1.5 mg/L.

Material and methods

This work was performed for the purpose of setting up a method already used routinely by POURQUERY Laboratories. The analyses were made on an ICP-AES VARIAN 730 ES, notably composed of a cyclonic spray chamber, a Meinhard nebulizer and a CDD detection system.

First, several rays per element were chosen in order to calculate limits of quantification on each ray by ten injections of acetic acid (4% in water). The analysis conditions are listed:

- Plasma power: 1.35 kW
- Plasma argon flow rate: 16.5 L/min
- Auxiliary argon flow rate: 1,50 L/min
- Sample uptake rate : 0.85 L/min
- Integration time : 20 s
- Peristaltic pump speed : 18 t/min

Only the rays which respected the standard were selected. To validate this method, several parameters were tested such as : linearity, repeatability and reproducibility. To this end, a calibration range (from 0.02 mg/L to 5 mg/L) for all elements was performed to quantify any sample. At the same time, high purity 10 000 mg/mL ICP standards were used to prepare four working standards. Four assays with ten repetitions were carried out to verify the repeatability and the reproducibility of the system.

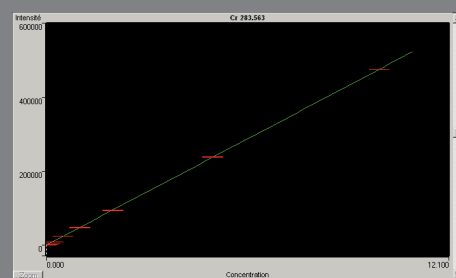
Results and discussion

Three calibration curves were built and the most linear was chosen for the routine method. An example of a calibration curve is presented in Figure 1. The different assays demonstrated that the method was repeatable and reproducible. Indeed, control cards of working standard were established and proved the efficiency of the method.

Conclusion

In conclusion, this technique can be validated to quantify Cd, Cr and Pb in aqueous extracts of ceramics. POURQUERY Laboratories will soon be audited for this method by COFRAC (Comité Français d'Accréditation).

Figure 1 : Calibration curve of Cr 283,563 nm



Laboratoire POURQUERY
93, Boul. du Parc d'Artillerie
69007 Lyon