

# **Experimental Planning for Validation (Collaborative Study in Interlaboratorials) of Methods for Detection and Quantification of Frauds and Impurities in Roasted and Grounded Coffee**

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## **SUMMARY**

This paper describes the planning and execution of tests with the purpose of carrying out experiments for validation of methods for the detection and quantification of frauds and impurities in roasted and grounded coffee. Another purpose is to obtain estimative of parameters of some requirements for validation to be used as reference for comparisons between methods. The contamination of the samples (Figure 1) was provided by the company Toko Industry Trade Exports and Imports Ltd., member of the ABIC (Brazilian Association of Coffee Industry), using concentrations determined by previous researches in barks and sticks. Barley, the most commonly raw material used in frauds in roasted and grounded coffee, and “açai”, the raw material used nowadays as an alternative fraud, with low level of differentiation when compared to coffee, were also used as contaminants. The material was packed in sealed envelopes. Three experiments were planned for validation, one by collaborative study (experiment of accuracy) and two others by interlaboratorial study. The imaging method was used in the collaborative study while all the other methods were used in the interlaboratorial validation. The aim of this work was to compare the accuracy of the three methods and verify the existence of the following aspects: a) scientific basis of the test methods b) the presence or absence of repeated habits in all methods c) probability of false negative / positive. An interlaboratorial experiment was conducted by the Equifarma using analysis by the conventional method. The Embrapa Scientific Instrumentation was responsible for the analysis using photothermic method in another interlaboratorial experiment. Another study was done by ITAL, Embrapa/CTAA, Embrapa/Cerrados using the method of measurement by images in collaborative study. In all three experiments distributions of probability hipergeometric were used for making a system random sample with no replacement, and uniform, for coding portions of the sample to be measured, which were generated in statistical program. The experiment of accuracy followed the series of precision ISO 5725-1 to ISO 5725-6, and the interlaboratorial were carried out following the description of NATA TN 17 and EURACHEM (1998). The sample portions were large in order to avoid the necessity of replacement in case of a total loss. In the experiments the analytical capacity of the laboratories in each method were considered, and each portion sample was coded with 3-digit randomly, reducing the incidence of trends of analysts (Figure 2). For the 5 laboratories a total of 438 units were prepared and also packed in sealed envelopes. Letters with instructions, number of units to be measured, enforcement order, and way of measuring results were sent to the laboratories.