

Validation and quality assurance applied to goat milk chemical composition: Minerals and trace elements measurements

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Abstract – In the present study, quality assurance programmes were implemented to validate and control the analytical methodologies used for the characterization of minerals and trace elements in goat milk from Portuguese breeds. With the exception of chloride that was determined by potentiometric titration, all the other elements were determined by spectroscopic techniques after different sample decomposition: P was measured by ultraviolet-visible molecular absorption spectrometry, Ca, Fe, K, Mg, Mn, Na and Zn by flame atomic absorption spectrometry and Cd, Co, Cr, Cu, Mo, Ni and Pb by electrothermal atomic absorption spectrometry. The methods performance characteristics, namely specificity, limit of detection, limit of quantification, working range, precision and trueness were evaluated. Measurement uncertainty was expressed in terms of precision and trueness. Precision under intralaboratory reproducibility conditions was estimated from triplicate analysis, and the trueness component was estimated in terms of overall recovery using either skim milk powder certified reference materials or spiked samples. The results obtained are discussed on the basis of the performance criteria required by EC regulations to verify when a method is suitable for food control. The methods used for the characterization of minerals and trace elements in goat milk complied with EC requirements since there was no matrix influence, the Horrat values were < 2.0, recoveries were within the interval 1.00 ± 0.10 for minerals and 1.00 ± 0.20 for trace elements and the combined uncertainty of the results were lower than the maximum standard uncertainty calculated using the uncertainty function approach. In relation to the limits of detection and quantification, the limits obtained for Pb were lower than those specified by EC regulation.

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