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Food Analysis and Method Validation

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Knowledge of the chemical composition of foods is the first essential in dietary treatment of disease or in any quantitative study of human nutrition. Health researchers and epidemiologists use nutrient intake studies to correlate food components with causes or prevention of disease. Dietitians counsel patients in dietary changes based on an analysis of their dietary habits. Food manufacturers determine the nutrient content of their products for food labels. Food service managers plan menus for schools, hospitals, and other institutions based on their nutrient content. Food composition data and information are used in these ways and collected from various sources (food industry, university, research institutes, etc). Therefore, the composition data should represent estimates of means and variability for selected foods, nationally representative of food supply, high quality analytical data, and other statistical parameters. There are many methods used for food composition analyses. If an analytical method is used at the national level for enforcement purposes, it must be properly validated. The ideal validated method is one that has progressed through an extensive collaborative study in accordance with international harmonized protocols for the design, conduct and interpretation of method performance studies. Method validation is the process used to confirm that the analytical methods employed for a specific test is suitable for its intended use. USP listed "Eight Steps of Method Validation". ICH divided the "Validation Characteristics" somewhat differently. The differences in the USP and ICH terminology is that ICH treats system suitability as a part of method validation, whereas the USP treats it in a separate chapter. Common parameters used for method validation are listed. "Accuracy" indicates the closeness of agreement between the value which is accepted either as a conventional, true value or an accepted reference value. "Precision" is the measures of the degree of repeatability and reproducibility of an analytical method, normally expressed as %RSD for a significant number of samples. Repeatability is the results of the method operating over a short time interval under the same conditions. Intermediate precision is the results from within laboratory variations. Reproducibility refers to the results of collaborative studies between laboratories and includes SD, RSD, CV, and CI. "Specificity" indicates the ability to measure specifically the analyte of interest in the presence of other components that may be expected to be present in the sample matrix. It measures the degree of interference from such things as other active ingredients, impurities, and degradation products. ICH divides "specificity" into identification and assay/impurity tests. "Identification" is demonstrated by the ability to discriminate between compounds of closely related structures, or by comparison to known references. "Assay and impurity tests" are represented by resolution of the 2 closest eluting compounds. "LOD" is defined as the lowest concentration of an analyte in a sample that can be detected, not quantitated. It is a limit test checking whether or not an analyte is above or below a certain value. "LOQ" is defined the lowest concentration of an anlayte in a sample. For better precision, a higher concentration must be reported and an appropriate number of samples should be analyzed. "Linearity" is the ability of the method to elicit analysis results that are directly proportional to analyte concentration within a given range. "Range" indicates the interval between the upper and lower levels of analyte. For assay, the minimum specified range is from 80-120% of the target concentration. For an impurity test, it is from the reporting level of each impurity to 120% of the specification. "Ruggedness" is the degree of reproducibility of the results obtained under a variety of conditions. It includes different conditions under different laboratories, analysts, instruments, reagents, days, etc. "Robustness" is the capacity of a method to remain unaffected by small delicate variations in method parameters. These all parameters should be validated before their introduction into routine use, whenever the conditions (samples matrix, instruments, etc.) change for which the method has been validated, and whenever the method is changed.

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