

## Techniques that allow the laboratory to validate its own performance within or between other laboratories

Techniques	Within laboratory validation	Between laboratory validation
Recovery study	x	
Replicate determination	x	
Use of standard and authentic samples	x	
Use of normal/routine samples with varying concentrations	x	
Use of food samples analysed by different methods	x	x
Analysis carried out by second analyst	x	x
Collaborative studies		x

### Recovery study:

When a constituent is available as a well-characterized material with known purity, it is possible to conduct recovery studies in which a defined amount of the constituent is added to portions of the food being analysed. Analysis of the food alone and with the added constituent can be used to calculate recovery of the added constituent (or “spike”). If a range of additions are made, effects of concentration can be measured.

### Replicate determination:

Both precision and accuracy are assessed by means of replicate assays on portions of the same food sample (which are assumed to be stable and identical regarding the quantity of analyte being investigated). In statistical terminology, the replicate results are considered as random samples from a hypothetical population of replicates; the mean (as well as other measures of location or central tendency) of these samples reflects the performance of the method with respect to accuracy, and the standard deviation (as well as other measures of dispersion) reflects its precision.

Duplicate analyses are normally the minimum required for food composition studies.

### Use of standard and authentic samples:

Ideally, standards would be prepared containing known amounts of the constituent of interest, in the same physicochemical form and in a similar food matrix to the one to be analysed. Clearly this ideal is virtually impossible to achieve, but various substitutes are available for use as standards.

Reference materials (RMs) and standard reference materials (SRMs) support accurate and compatible measurements by certifying and providing samples with well-characterized composition. These materials are used to perform instrument

calibrations as part of overall quality assurance programmes, to verify the accuracy of specific measurements and to support the development of new measurement methods.

**Use of normal/routine samples with varying concentrations:**

If an analysis is to be carried out on a substrate that is new to the laboratory, the selected method should be applied to a series of routine food samples containing a fairly wide range of the constituent of interest. If such a series is not available, a set should be prepared by the careful blending of known amounts of the constituent with a food sample of known composition. Direct addition of small quantities of a constituent to large weights of a food should not be attempted; low levels should be obtained by serial dilution, preferably starting with a solution of the constituent.

**Use of food samples analysed by different methods:**

When introducing an unfamiliar or new method, it is helpful to re-analyse food samples that have previously been analysed by another, established method. Such samples should be analysed by replicate determinations, and then re-analysed after accurate dilution with some inert material such as water, oil or sand. If replicates and differences between samples are satisfactory, it is usually safe to proceed.

**Analysis carried out by second analyst:**

The best kind of check analysis is for a second, preferably more experienced analyst to repeat the analysis by the same method on a separate portion of the same analytical sample. A preparation of a new analytical sample from the original food sample is recommended, because it permits estimation of error introduced during preparation of the analytical sample.

Another useful check involves calculations and analyses by a second person, because of the high frequency of mathematical errors and simple mistakes.

**Collaborative studies:**

Three major types of collaborative studies exist, that either provide comparative assessments of laboratory performance, are used to establish the performance of a method, or used for the development of standard certified materials.