

TEST METHOD VALIDATION

What is Method Validation?

Method validation is defined as the confirmation, through the provision of objective evidence, that the requirements for a specific intended use or application have been fulfilled. The aim is to establish the operational limits and performance characteristics of a new, modified or otherwise inadequately characterised test method.

When is it done?

Test methods should be validated when:

- a new test method is being developed
- an established test method is modified
- QC indicates that an established method is changing with time
- demonstrating the equivalence between two methods, e.g. a new method and a standard method.

How is it done?

The experiments used to determine method performance characteristics should be conducted with equipment that is within specification, working correctly and adequately calibrated. The analyst carrying out the experiments must be competent in the field of study and capable of making appropriate decisions from the data produced during the study.

The following are common characteristics tested during method validation studies:

- Specificity/selectivity – ability to accurately measure the analyte in the presence of interferences.
- Linearity - ability of an analytical method to produce test results which are proportional to the concentration of analyte in samples within a given concentration range.
- Accuracy – closeness of the measured value to the true value for the sample.
- Range – the concentration interval over which acceptable accuracy, linearity and precision are obtained.
- Precision (Repeatability & Reproducibility) – the amount of scatter in the results obtained from multiple analyses of a homogeneous sample.
- Detection Limit – lowest concentration of the analyte that can be confidently detected by the method.
- Quantization Limit – strictly the lowest concentration that can be determined with an acceptable level of repeatability, precision and trueness.
- Robustness – ability of the test method to remain unaffected by small but deliberate changes, e.g. temperature.
- Recovery – assesses the efficiency of the method in detecting all of the analyte present.

Why is it necessary?

- To provide objective evidence that the results are accurate and reliable
- To demonstrate that a test method is “fit for purpose”
- Validation data can be used to design QC programs