18.7.2 Operational terms

Test Sample/Analytical Sample/Test Material

The homogeneous, stable material with a certain property, a specified composition, or containing one or more test components at given concentrations in a defined matrix that is subdivided into identical portions sent to the laboratories participating in an interlaboratory study.

Notes:
(1)   The homogeneous, stable parent substance prepared by the organizer of the interlaboratory study may be designated as the "material" and the subdivided portions may be designated as the "test sample", if it is necessary to distinguish between them. The portion removed from the test sample is the "test portion". If only analytical chemistry is involved, the term "analytical" may be substituted for "test".

(2)   Although it is preferable that the composition of the test component be known by formulation, independent analysis, or by assignment so that recovery (or bias, trueness, systematic error) may also be measured, this is not always possible at trace levels, or with natural products, tissues, sediments, sludges, or environmental matrices such as waters and wastewaters, or with analytes that are defined by the method, e.g., moisture, boiling range, etc.

Matrix

The carrier of the test component (analyte); all of the constituents of the material except the analyte; or the material with as low a concentration of the analyte as is possible to obtain.

Notes: Some interlaboratory studies will include the submission of a blank matrix. The instructions should then specify how to report low-level values. The preferred reporting procedure with instrumental methods applied to blank materials is to translate the signal into the corresponding concentration through the calibration graph. The resulting apparent concentration should be reported as it appears -positive, negative, or zero. Qualitative terms such as "less than", "below the detection limits of _ units", assigned values of zero, or a multiple of the detection limit, e.g., 0.5, $\sqrt{2}$ etc., x the detection limit, should not be accepted. An uncertainty statement must be given.

Laboratory

The place with physical facilities and an environment in which the analyst(s) operates to gather data for the interlaboratory study.

Notes:

(1)   Although analytical results are ascribed to a laboratory, they usually reflect the output of an analyst or a team of analysts. When the proficiency of individual analysts within a laboratory is to be evaluated, provision for independent operation by each of the analysts must be specified in the study protocol.

(2)   Separate laboratories of a single organization with independent facilities, and with different local management, instruments, and calibration materials, are treated as different laboratories.

Data Set

The group of estimated values of quantity or concentration, final results, or decisions (yes/no; accept/reject; present/absent) from the group of participating laboratories in an interlaboratory study of a specific material, at a specific level.

Note: The term "assay" has also been applied to this concept - the data set from a given matrix/analyte level/method combination. The use of "assay" should be confined to the operation of analysing the material.

Determination

The complete analytical (test) operation starting from the removal of the single test portion to reporting the final result.

Notes:
(1)   The purpose of this definition is to provide a measure of the amount of work required to conduct the interlaboratory study.
(2)   A determination (an operation) must be distinguished from a final result (a datum or estimate). Sometimes the average (mean) of the results from several replicate determinations or independent determinations is the final reported result.

Replicate

Each of the set of multiple determinations conducted on identical test portions from one test sample, by one laboratory by the same method and protocol.

Notes:
(1)   To avoid ambiguity, the term "replicate" or "replication" used alone should be employed only in the context of measurement (analysis) and not in the sense of "preparation of multiple units" or collect "replicates" unless the usage is explicit, e.g., "Prepare replicate test samples from the laboratory sample".

(2)   The analyst may or may not be aware that a test sample is a replicated material. If the determinations are conducted concurrently (regardless of knowledge of the identity of the test samples), the reported results can provide a measure of within-run (-batch, -group) precision, usually designated as repeatability precision. This type of measurement replication often provides an over-optimistic estimate of within-laboratory variability. Consequently, if the identity of the replicate test samples is disclosed, it is better to request the replicate analyses to be conducted at different times in order to obtain a more realistic estimate of within-laboratory variability. If the replication is conducted at different times, the reported values can provide an estimate of *between*-runs precision, which includes *within*-run precision. The between-runs precision parameter is intermediate between the repeatability and reproducibility (among laboratories) precision.

(3)   Replication from the very beginning of the removal of the test portion provides an estimate of repeatability. Repetition beginning at any later stage (e.g., aliquots from the same dissolved test portion) does not provide an estimate of repeatability since the variability introduced by the omitted steps is not included in the final measurement. Presenting a test solution repeatedly to an instrument provides an estimate of instrumental precision only. Instructions and reports should be very clear as to which readings are to be reported separately and which are to be combined.

(4)   The final results from each of the series of measurements may be called a "replicate value", indicating exactly which item or step was replicated.

Protocol

The detailed set of instructions describing the design, conduct, and reporting of a study, test or trial.

Notes:
(1)   The protocol of an interlaboratory study should specify the minimum number of laboratories, the number and nature of the test samples, the details of the method(s) of analysis, and the number of replicate determinations to be performed, as applicable. It should also contain practical details of transport, receipt, and preservation of test samples, the performance of the statistical analysis (particularly the outlier removal techniques), and the reporting of final results.

(2)   To avoid misunderstandings, the protocol must be completely specific about which readings are to be reported separately and which are to be combined (averaged), and whether reported replicate results apply to the complete determination or only to a portion of it. The statistical analysis must be consistent with the instructions to the participating laboratories.

(3)   Participants should not provide more or fewer data than requested, and should report the same number of significant figures. These requirements simplify the subsequent statistical analysis.