

18.3 Sampling

18.3.1 Hierarchy of terms used in sampling

The primary material delivered to the laboratory is the *laboratory sample*. If it is homogeneous, a portion may be removed directly for weighing, measuring, or direct analysis as the *test portion*. If the laboratory sample requires preparation (subdividing, mixing, drying, reduction in particle size, etc.) the prepared material is the *test sample*, from which a test portion is removed for analysis. Alternative terms which may be used when only analytical chemistry is involved are *analytical sample* and *analytical portion*. However, since other than chemical examinations may be conducted by the laboratory (e.g., physical, biological, sensory), "test" is more appropriate for the general case as applied to the sample and portion. If the test portion or analytical portion is dissolved, directly or by reaction, as preliminary to further operations, the resulting solution is the *test solution*. When further reactions, separations, or operations are conducted on the test solution, the final solution used for measurement is the *treated solution* or the *measurement solution*. Very often the analytical literature, particularly descriptions of methods of analysis, designates the chemical operations between the taking of the test portion and the measurement of the characterizing property as "preparation of sample." This usage is incorrect since once the test portion is measured, all subsequent operations are analytical in nature. These chemical operations should now be described as "Preparation of the Test (or Measurement) Solution", "Separation", "Isolation", or "Purification" [of analyte(s)] procedures. In no case should the term "sample" or its derivatives be used since this would suggest an operation at stages prior to removing the test portion, e.g., the preparation of a test sample from a laboratory sample.

Although, strictly speaking, the laboratory report describes the composition or properties of the test portion, the results are usually taken to apply to the lot. Actually, the results should be extrapolated only to the highest level under the control of the analyst, generally the laboratory sample. Only if replicate test samples have been prepared from the laboratory sample can the sampling uncertainty at this stage be estimated, although method performance variability will always be a confounding factor. Similarly, the uncertainty involved in extrapolating from the laboratory sample to the parent lot can be estimated only if replicate lot samples have been taken, prepared, and analyzed. Usually the uncertainty involved in the extrapolation from the test portion to the lot is resolved by assuming sampling to be representative at all stages of sampling and reduction. If there is no basis for estimating the sampling error involved in the extrapolation (historical information is often satisfactory), the laboratory report should make this point clear.