Pure & Appl. Chem., Vol. 62, No. 6, pp. 1193–1208, 1990.
Printed in Great Britain.

INTERNATIONAL UNION OF PURE
AND APPLIED CHEMISTRY

ANALYTICAL CHEMISTRY DIVISION
COMMISSION ON ANALYTICAL NOMENCLATURE*

NOMENCLATURE FOR SAMPLING IN ANALYTICAL CHEMISTRY

(Recommendations 1990)

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Nomenclature for sampling in analytical chemistry (Recommendations 1990)

Abstract

The IUPAC definition of the term SAMPLE appearing in the "Compendium of Analytical Nomenclature, Definitive Rules 1987" is, "the actual material investigated, whether diluted or undiluted." Although many colloquial analytical chemical communications use this definition, it frequently results in confusing, ambiguous, and contradictory usages. The term has been used in at least 17 ways in IUPAC documents and other scientific literature. Such ubiquitous applications often do not permit analysts to orient themselves as to where they are in the sampling and analytical scheme. A remedy is proposed, based upon the vocabulary of the International Organization for Standardization (ISO), that requires:

(1) Confine the use of the term SAMPLE to its statistical concept: If a potential exists for a sampling error due to the heterogeneity of a population, the term SAMPLE with an appropriate modifier to indicate its position in the sampling scheme should be used (i.e., increment, primary sample, secondary sample, etc., laboratory sample, test sample, test portion).

(2) Once analytical work begins by measuring the test portion, avoid the term SAMPLE; use TEST or UNKNOWN as the modifier as in TEST SOLUTION, TEST EXTRACT. For the common step usually designated as PREPARATION OF SAMPLE in methods of analysis, use more specific terms, such as PURIFICATION, SEPARATION, or ISOLATION [of analytes].

(3) Use the term SPECIMEN for a sample from a parent population that changes with time, as in living organisms, circulating blood, or a flowing river. In this case the phenomenon under study and the sampling error are confounded in such a way that they can not be separated.

CONTENTS

1.0 Introduction
1.1 Scope
1.2 Use and misuse of the term "Sample"
Other problems
1.3 Special relationships [Analysis of the parent population]
1.4 Static vs. dynamic systems [The specimen problem]
1.5 The sample unit
1.6 Bulk sample [Sampling bulk vs packaged materials]
1.7 Hierarchy of terms [Laboratory sample -- Test sample -- Test portion -- Test solution]
1.8 Point where sampling ends and analysis begins

2.0 Definitions
2.1 General terms
2.2 Material units
2.3 Sample types
2.4 Sample preparation [Laboratory sample to test sample]
2.5 Sampling stages

Index

1.0 INTRODUCTION

1.1 SCOPE

This document is intended to furnish concepts, terms, and definitions in the field of sampling that are relevant to analytical chemistry and that are generally applicable regardless of what sampling objective, material, location, quantity, or form is involved. Although almost every specialized area of analytical chemistry has developed its own nomenclature, the intent here is to permit any specialized system to be able to relate its semantics and notation to some aspect of this document, so that there can be compatibility in concepts, if not entirely in substance. As far as possible, terms have been defined within a practical framework. In this introduction the term "lot" is used in a very general sense as the parent population which is to be sampled.

There are two aspects to sampling: design and implementation. In the design phase the application of statistical principles is expected to supply a statistical sampling plan to determine the acceptability of a lot, the magnitude of a property, or the quantity of a constituent, within a specified degree of variability with a stated degree of confidence. The statistical sampling plan results in a specification of the size, number, and location of portions to be removed from a lot. A statistical sampling plan is intended to minimize the difference between the properties as estimated from a sample and the actual properties of the lot, and to limit or control the uncertainty generated by the sampling operation, all within practical constraints.

The implementation phase of sampling involves the physical realization of the statistically designated portions and the removal and preparation of them -- an operation easier to state than to perform. This aspect also requires consideration of the tools of sampling, the containers used, preservation of the portions removed, and precautions necessary to avoid changing the characteristics of the sample (sterility, contamination, moisture loss or gain, etc.).

In many cases, the analytical chemist has no responsibility for either the design or implementation aspects of sampling. His first contact with the operation is usually the receipt of a laboratory sample. Yet he is often expected to characterize a lot on the basis of a portion over which he had no control. Nevertheless, the analytical chemist must have a perception of the uncertainty involved in sampling, since this determines to a large extent the variability that can be tolerated in the analysis, when the analytical methodology is not the limiting factor. Knowledge of the potential sampling error is important since if the sampling error is already more than about 2/3 of the total error, reduction in analytical error is of only marginal importance.

1.2 USE AND MISUSE OF THE TERM "SAMPLE" IN ANALYTICAL CHEMISTRY

The term "sample" as used in analytical chemistry should be applied exclusively to represent a portion of material selected in some manner to represent a larger body of material. The result obtained from the sample is merely an estimate of the quantity or concentration of a constituent or property of the parent material. The parent material may be homogeneous or heterogeneous as well as static or dynamic. The use of a sample always introduces an uncertainty, arising from heterogeneity of the parent material, in extrapolating from the smaller portion to the larger portion -- the "sampling error."

Colloquially, the term "sample" and its derivatives frequently have been used to designate the portion being analyzed at various stages in the analytical procedure as distinct from a standard and a blank which may accompany it. The present IUPAC definition (Compendium of Analytical Nomenclature Definitive Rules 1987. Henry Freiser and George H Nancollas, Second Edition Blackwell Scientific Publications Oxford, UK, p. 39), which is found in the section on thermogravimetry is, "The sample is the actual material investigated, whether diluted or undiluted." *This usage, although quite common in IUPAC* documents and other scientific communications, is not consistent with the proposed concept since it can result in inconsistencies and ambiguities as illustrated in some of the examples given below. The current practice should be discouraged, particularly when other terms, e.g., "test" or "unknown," are available for the desired expression. In the following examples, square brackets enclose the term(s) that the word "sample" was intended to mean.

1.2.1 Material in general E.g., "organic samples [materials]; mineral samples [materials]; hard samples [materials, substances, products, commodities, items, etc.]."

1.2.2 Analyte The component measured, e.g., "The sample [analyte] is transformed into a metal vapor." "Sample [analyte] enrichment is performed by evaporation."

1.2.3 Quantities A specific mass or volume, e.g., "Samples [quantities] as large as 500 mg . . . ."

1.2.4 Aliquot A known fractional portion of a homogeneous mass, e.g., "Remove a 20 mL sample [aliquot] from the 100 mL volumetric flask."

1.2.5 Phase The physical form of material, e.g., "The final sample [phase] presented to the instrument may be a solid, liquid, gas, or solution."

1.2.6 Test portion The actual material weighed or measured for the analysis, e.g., "Accurately weigh a 5.00 g sample [test portion]."

1.2.7 Test solution The solution obtained by dissolving the test portion, e.g., "Transfer the sample [test] solution to the distillation flask."

1.2.8 Treated solution The solution used for the measurement of a property related to the original analyte, e.g., "Determine the absorbance of the sample [treated] solution . . . ."

1.2.9 Stream splitter E.g., "The volatile analyte is supplied directly to the sample source [stream splitter]."

1.2.10 Injection loop E.g., "The solution was applied to the column through a sample [injection] loop."

1.2.11 Specimen A specifically selected portion taken as representative of the parent material at the time it is drawn, e.g., "A sample [specimen] of blood was obtained from the patient."

1.2.12 Path length The dimension of the cell or cuvet involved in the measurement of absorbance (or other property), e.g., "b = sample thickness [path length] in cm."

1.2.13 Derived from a sample (adjective) E.g., "sample solution" [solution of a test portion; test solution]; "sample components" [analytes].

1.2.14 Removal of a portion (verb) E.g., "The laboratory sample is subsampled [subdivided] to produce a test sample."

1.2.15 Determination The entire analytical operation on a single test portion from the measurement of the test portion to the calculation of the analyte content, e.g., "The new method reduces analytical time by 0.5 hour per sample [determination]."

1.2.16 Matrix E.g., "Determine baseline of blank sample [matrix without analyte]."

1.2.17 Unknown test material E.g., "In spite of the limited quantity of sample [test sample], low resolution mass spectrometry was conducted."

In many cases, the meaning is clear from the context, but this is not always the case. A vocabulary has been developed, based upon the International Organization for Standardization (ISO) formulations (ISO 3534-1977; ISO 3534 (Revision); ISO 7002-1986; International Organization for Standardization, Geneva, Switzerland; also available from national standardization organizations), which removes much of the uncertainty and ambiguity in meanings, and which permits orientation as to what stage of sampling or analysis is being discussed.

1.3 SPECIAL RELATIONSHIPS BETWEEN THE PORTION EXAMINED AND THE PARENT MATERIAL

Sometimes sampling can be accomplished without taking discrete portions of the parent material or lot. The following sections provide some examples where the need for sampling is eliminated by examining (a) the entire lot, material, or population, (b) a selected portion of it, or (c) a portion so large as to make the sampling error negligible. When the fraction examined is an appreciable part of the parent population, a modified statistical treatment based on "sampling without replacement" is required.

1.3.1 Large-scale direct examination or analysis of homogeneous parent materials by electromagnetic radiation techniques (e.g., radar, microwaves, infrared, X-rays), or by acoustics.

1.3.2 Processing of a major fraction, or the entire product. In some of these cases, particularly where the analyte(s) is present at very low concentrations, e.g., air, water, ores, etc., the analyte may be concentrated by procedures such as adsorption, condensation, electrostatic precipitation, filtration, flotation and other pretreatment techniques. The isolated material, which usually has a quantitative relationship to the parent material, often is considered and described in terms ordinarily applied to the parent stage, e.g., gross sample, secondary sample, laboratory sample, test sample, and test portion. The isolated material also may be designated as a "modified sample." The collection of whole blood, but the analysis of the plasma is a similar case.

1.3.3 Sampling by direct examination of the parent material in situ (e.g., pH of soils, infrared reflectance analysis of painted surfaces). In these and similar cases, it may not be recognized that the instrument is performing its own sampling on a micro scale. A determination of bulk composition, even on the basis of numerous micro samples, involves considerable extrapolation with a resulting potential for relatively high sampling error.

1.3.4 Directed or focused sampling (not necessarily representative or random sampling) or removal or isolation of portions of the parent material by separation, concentration, or selection techniques (e.g., by use of magnetic, density, adsorption, or optical properties) and examination of the isolate, condensate, adsorbate, individual particles, surfaces, and/or profiles of the parent material itself or of the laboratory sample (e.g., microprobes). In food analysis, for example, a deliberate attempt is made to locate the specific adulterated portion of a lot, undiluted by sound material.

1.4 REMOVAL OF PORTIONS FROM A STATIC VERSUS A DYNAMIC SYSTEM

Many situations requiring sampling and chemical analysis involve static conditions, i.e., the composition of the parent material can be considered as permanent with respect to position in space and stable in time. The opposite condition is commonly encountered in environmental and clinical chemistry, where the parent material is almost always changing with respect to time (dynamic) and the removal of a portion of the parent material at any instant reflects only a state at that time and at a particular site. In sampling static lots, time is irrelevant and the variable is the sampling position in the space occupied by the lot.

If the purpose of sampling (removing a portion to represent a larger body) is to reflect the change in composition of the parent material with time (monitoring) or the average composition over time, then the sampling plan must provide for the removal of physically comparable units (or increments) at each "sampling" time. In these instances, as with blood or urine in clinical chemistry where the unit removed is known as a specimen, or as with a flowing river or the point of air "sampling" in environmental chemistry, the variable is time and the sampling positions should be constant in space. In one sense, the specimen is a convenience sample (2.3.5).

In sampling under dynamic conditions, often the major variation is not heterogeneity; rather it is fluctuations of the phenomenon itself with time. Such studies are conducive to continuous or systematic sampling or to in situ testing. Usually the portion removed ("the sample") from the parent material is an exact (aliquot) or a fair (specimen) representation of the parent material that exists at a given time and in a given space. The fact that it can never again be reproduced (as in a flowing stream or circulating blood, or in the atmosphere, ocean, or bodies of nonflowing fluids subject to temperature, pressure, or concentration gradients) presents difficulties in applying statistical control and consequently cannot be the subject of conventional statistical sampling plans. Nevertheless, in the vocabulary of sampling, the specimen of clinical chemistry (and the corresponding portions of environmental chemistry) would be considered the laboratory sample, test (analytical) sample, or the test (analytical) portion. Which of these expressions would be appropriate depends on whether or not further manipulation, such as homogenizing, taking an aliquot, etc., is required before the analytical determination is performed.

Conditions exist which require a combination of both time and space sampling; e.g., in the enforcement of maximum limits of air pollution, where any portion removed exceeding a specification may result in legal or economic penalties. Often a better representation of a static lot can be obtained by periodic sampling of the material as it is moved into or out of storage. This special case of dynamic sampling can be differentiated from the nonreproducible cases outlined above since the lot is finite and movement in space does not change the overall composition of the parent lot.

1.5 THE SAMPLE UNIT

Each single portion (unit) that is removed from a bulk lot is called an "increment." It is usually taken with a trier, bucket, shovel, etc. When this term "increment" is encountered by chemists for the first time in the sense of a sampling unit from a bulk lot, it appears to be an incorrect application of a word meaning "an addition." Yet it is a well-established term in the sampling literature since it is an incremental portion to be either grouped or combined with other incremental portions to form the primary sample. It is not considered as an incremental portion of the lot. Similarly, the use of "segment" as a large, partially packaged portion of a bulk lot is also an unusual application of this term, which is well- established in the sampling literature. An increment from a bulk lot is analogous to a unit from a packaged lot. When the units from a packaged lot are large (e.g., 10 pound or 5 kg bags of sugar), increments may be removed from them. In clinical chemistry, the unit removed from the lot (body) is called the specimen.

1.6 SAMPLING BULK MATERIALS AND PACKAGED MATERIALS (see figure)

An important concept which creates difficulties in sampling nomenclature is the considerable difference in the mechanical and statistical concepts of sampling bulk materials on the one hand and sampling packaged materials on the other.

Bulk materials may consist of a single pile (coal, fertilizer), a plot of ground (soil), a shipload of grain, or a portion of the atmosphere or ocean, all characterized by a lack of permanently identifiable units. For convenience in handling, bulk materials may be partially packaged in smaller units called segments (bags, bales, drums) separated in space or in time (moving on a conveyor belt), which can be identified for random sampling. In bulk materials, the segments are usually still too large for submission as a laboratory sample, so in practically all cases of sampling bulk materials, the initial physical sample is called an increment, which is a unit created by the sampling device (e.g., trier, shovel). The increments may be combined (composited) to obtain a physically averaged sample (gross sample; bulk (bulked) sample; lot sample; composite sample), or they may be combined into several composite samples (partial samples), or the individual increments may be analyzed separately. In the case of the single composite, no estimate can be made of the heterogeneity of the parent material; in the other cases (analysis of several composites or individual increments), an estimate can be made of heterogeneity. In practically all cases of sampling bulk materials by using increments or composites, the primary sample as well as the laboratory sample must be reduced in size (subdivided by riffling, or rolling, coning and quartering, etc.) to a test (or analytical) sample. Often this reduction in physical size must be accompanied by a reduction in particle size as well. At some stage, a final sample may be produced for possible subdivision into presumably identical portions for distribution to the parties involved, for submission to the laboratory for testing, for forwarding to the owner of the goods, and for storage for use in case of arbitration or dispute. Such a final sample may be prepared in the field or in the laboratory.

Packaged goods are comprised of identifiable units which may be assigned numbers, either actually or conceptually. Numbered units are then selected for the laboratory sample by use of a table of random numbers. This technique may be used even though the units may be further packed in larger and larger multiple units (cases, pallets, truckloads), and even though it is often impractical to obtain the actual units selected in this manner. In theory, at least, the selection of units for the sample of packaged materials may be performed by a statistically designed random selection procedure. Random selection of units from bulk materials may not be possible because of unrecognized shifting of portions of the parent goods. The physical act of removing increments from solid bulk materials changes the location of particles in the neighborhood of the sampling point.

Because of the sheer quantity of transported and warehoused material in the modern economy, much sampling is performed as "convenience sampling" -- removal of accessible portions from the lot. Such action results in a larger uncertainty in subsequent test estimates than would occur if random sampling were performed.

1.7 USE OF THE TERM "BULK SAMPLE"

The use of the term "bulk" in the sense of "to combine" as well as in the sense of an undifferentiated mass (unpackaged material) creates an ambiguity, since the term "bulk sample" can mean a sample from a bulk lot or a composite of units or increments. The latter usage is well established in the sampling literature. The substitution of "combine" as an alternative term (combined sample) or "lot sample" or "batch sample" for "bulk sample" in the sense of the quantity of material the sample is meant to represent is to be encouraged.

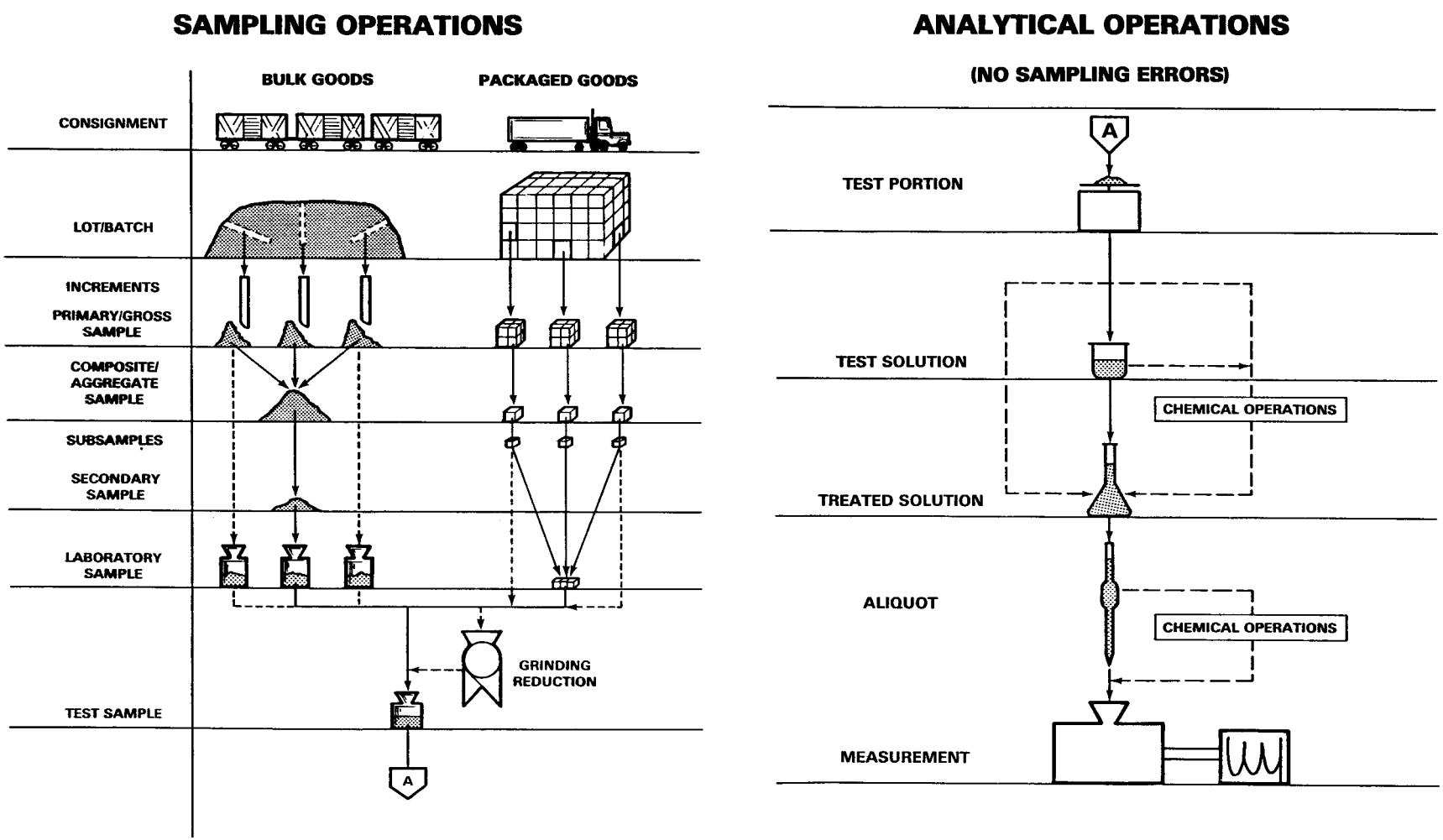

Figure 1. The relationships of the operations involved in sampling and analysis. The lower "A" of the sampling operations continues with the upper "A" of the analytical operations.

1.8 HIERARCHY OF TERMS (see figure)

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." By the proposed concept, 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.

1.9 THE POINT WHERE SAMPLING ENDS AND ANALYSIS BEGINS (see figure)

Sampling ordinarily ends with the removal of the test (or analytical) portion from the test (or analytical) sample. The weighing or measuring of this test portion is the first step in an analysis. Methods of analysis are usually so designed that any further subdividing (aliquoting) introduces negligible sampling error. By the concepts proposed in this document, the term "sample" or its derivatives should no longer be used once analytical operations on a homogeneous test portion have begun. (Exceptions can be visualized as, for example, an attempt to remove an aliquot from an unstable suspension of the test portion may introduce a sampling error. Such cases would be obvious and would be expected to be rare.)

2.0 DEFINITIONS

2.1 GENERAL TERMS

2.1.1 Sample

A portion of material selected from a larger quantity of material.

NOTE: The term "sample" implies the existence of a sampling error, i.e., the results obtained on the the portions taken are only estimates of the concentration of a constituent or the quantity of a property present in the parent material. If there is no or negligible sampling error (2.1.5), the portion removed is a test portion (2.5.7), aliquot (2.5.10), or specimen (2.2.6). The term "specimen" is used to denote a portion taken under conditions such that the sampling variability cannot be assessed (usually because the population is changing), and is assumed, for convenience, to be zero (see 2.2.6). The manner of selection of the sample should be prescribed in a sampling plan, 2.1.2.

2.1.2 Sampling plan

A predetermined procedure for the selection, withdrawal, preservation, transportation, and preparation of the portions to be removed from a population as samples.

NOTE: Summarizing the test values or observations from the selected portions yields an estimate for the concentration of an analyte or a value for a property determined with a calculable degree of uncertainty at a specified confidence level. A sampling plan includes the designation of the number, location, and size of the portions, and instructions for the extent of compositing and for the reduction (in amount and fineness) of the portions to a laboratory sample and to test portions. It may also contain acceptance criteria. Some sampling plans do not include more than instructions for the statistical selection of portions to be removed. Such plans should properly be designated as "statistical sampling plans."

2.1.3 Characteristic

A property or attribute of a material that is measured, compared, or noted.

NOTES: (1) Attributes are ordinarily qualitative characteristics, but quantitative characteristics (variables) may be converted into attributes by assigning items to certain categories on the basis of the measured values.

(2) The value of the characteristic determined as a result of an observation or test is the observed value; when determined by a specified test method, it is called the test result.

(3) The concentration or quantity of an analyte as estimated by use of a sample is usually the characteristic of interest in analytical chemistry.

2.1.4 Homogeneity, heterogeneity

The degree to which a property or a constituent is uniformly distributed throughout a quantity of material.

NOTES: (1) A material may be homogeneous with respect to one analyte or property but heterogeneous with respect to another.

(2) The degree of heterogeneity (the opposite of homogeneity) is the determining factor of sampling error.

2.1.5 Sampling error

That part of the total error (the estimate from a sample minus the population value) associated with using only a fraction of the population and extrapolating to the whole, as distinct from analytical or test error. It arises from a lack of homogeneity in the parent population.

NOTES: (1) In chemical analysis, the final test result reflects the value only as it exists in the test portion. It is usually assumed that no sampling error is introduced in preparing the test sample from the laboratory sample. Therefore, the sampling error is usually associated exclusively with the variability of the laboratory sample.

(2) Sampling error is determined by replication of the laboratory samples and their multiple analyses. Since sampling error is always associated with analytical error, it must be isolated by the statistical procedure of analysis of variance.

2.2 MATERIAL UNITS FOR SAMPLING

2.2.1 Consignment

A quantity of material transferred on one occasion and covered by a single set of shipping documents. It may consist of one or more lots or portions of lots.

NOTES: (1) The presence of different lots in a consignment is important from the point of view of the sampling plan and the interpretation of the results of analysis.

(2) The term "population" is used as the general term for the quantity of parent material being sampled when it is immaterial if the parent body is a consignment, lot, batch, entity, etc.

2.2.2 Lot

A quantity of material which is assumed to be a single population for sampling purposes.

2.2.3 Batch

A quantity of material which is known or assumed to be produced under uniform conditions.

NOTE: Some vocabularies assume "lot" and "batch" to be synonymous. The distinction made here with respect to knowledge of production history permits a lot to consist of one or more batches and is useful in interpreting the results of analysis.

2.2.4 Unit/item/portion/individual

Each of the discrete, identifiable portions of material suitable for removal from a population as a sample or as a portion of a sample, and which can be individually considered, examined, or tested, or combined.

NOTE: In the case of sampling bulk materials (or large packages), the units are increments, created by a sampling device. In the case of packaged materials, the unit may vary with the level of commercial distribution.

EXAMPLE: An individual piece of candy is the sampling unit at the consumer level; a package of individual pieces is the sampling unit at the retail level; a carton of packages is the sampling unit at the wholesale level; a pallet of cartons is the shipping unit at the distribution center level; and a truckload of pallets is the consignment unit at the manufacturers level. Before packaging, the bin containing the individual pieces, would be the bulk lot (or batch) for sampling.

2.2.5 Segment (applies to bulk materials)

Each of the single, large portions of material pre-existing either in space (e.g., bags, bales, drums) or accumulated during a fixed time (e.g., discharge from a conveyor belt) or formable as increments by a sampling device. Segments may be actual or conceptual.

2.2.6 Specimen

A specifically selected portion of a material taken from a dynamic system and assumed to be representative of the parent material at the time it is taken.

NOTES: (1) Although the specimen may not be reproducible in time, e.g., it may be taken from a flowing stream or a portion of blood, no separable sampling error exists since this error is unavoidably included with the corresponding error of the estimate of the property, function, or analyte being studied. A specimen may be considered as a special type of sample, taken primarily in time rather than in space.

(2) The term "specimen" has been used both as a representative unit and as a nonrepresentative (often better than most) unit of a population, usually in clinical, biological, and mineralogical collections. "Collections" in this case is used as either a noun or verb. This usage is almost always self-evident, and thus would not be confused with a time-type sample.

2.3 SAMPLE TYPES

2.3.1 Random sample

The sample so selected that any portion of the population has an equal (or known) chance of being chosen.

NOTE: Haphazard or arbitrary choice of units is generally insufficient to guarantee randomness.

2.3.2 Representative sample

A sample resulting from a sampling plan that can be expected to adequately reflect the properties of interest of the parent population.

NOTE: A representative sample may be a random sample or, for example, a stratified sample, depending upon the objective of sampling and the characteristics of the population. The degree of representativeness of the sample may be limited by cost or convenience.

2.3.3 Selective sample

A sample that is deliberately chosen by using a sampling plan that screens out materials with certain characteristics and/or selects only material with other relevant characteristics.

2.3.4 Stratified sample

A sample consisting of portions obtained from identified subparts (strata) of the parent population. Within each stratum, the samples are taken randomly.

NOTE: The objective of taking stratified samples is to obtain a more representative sample than that which might otherwise be obtained by random sampling.

2.3.5 Convenience sample

A sample chosen on the basis of accessibility, expediency, cost, efficiency, or other reason not directly concerned with sampling parameters.

2.3.6 Umpire sample/referee sample/reserve sample

A sample taken, prepared, and stored in an agreed upon manner for the purpose of settling a dispute.

NOTES: (1) The agreement usually extends beyond the sample to the basis for reaching a decision (e.g., quantity of material from which taken, use of a third party, and criteria serving as the basis for acceptance, rejection, or economic adjustment).

(2) The term "Reference Sample" has been used in this context but this term more properly should be used in conjunction with a "Reference Material" or "Reference Standard" which has a true or assigned value for a constituent or property. One of the characteristics of a reference material or reference standard is that it must have a negligible sampling error between test portions.

2.3.7 Replicate (duplicate) sample

Multiple (or two) samples taken under comparable conditions. This selection may be accomplished by taking units adjacent in time or space.

NOTES: (1) Although the replicate samples are expected to be identical, often the only thing replicated is the act of taking the physical sample.

(2) A duplicate sample is a replicate sample consisting of two portions.

(3) The umpire sample (2.3.6) is usually used to settle a dispute; the replicate sample is usually used to estimate sample variability.

2.3.8 Sequential sample

Units, increments, or samples taken one at a time or in successive predetermined groups, until the cumulative result of their measurements (typically applied to attributes), as assessed against predetermined limits, permits a decision to accept or reject the population or to continue sampling. The number of observations required is not determined in advance, but the decision to terminate the operation depends, at each stage, on the results of the previous observations. The plan may have a practical, automatic termination after a certain number of units have been examined.

2.3.9 Multistage sampling

Samples taken in a series of steps with the sampling portions constituting the sample (units or increments) at each step being selected from the larger or greater number of portions of the previous step, or from a primary (2.5.2) or composite sample (2.5.3).

NOTE: The first set of portions (units or increments) taken from the population available for sampling is the primary sample. The subsequent samples (secondary, tertiary, etc.) are the sets of subsamples, units, items, individuals, or increments taken from the preceding step. The units may be different at different steps of multistage sampling (e.g., pallets, cases, packages).

2.3.10 Combined sample

A sample obtained by removing specific fractions by separation or selection techniques (e.g., heavy liquid, magnetic, sieving, etc.), analyzing the fractions separately, and combining the results mathematically.

NOTES: (1) When not combined, the sample is a modified sample (2.3.11).

(2) This term should not be confused with composite sample (2.5.3).

2.3.11 Modified sample

A sample or a known fraction of the parent population in which the analyte has been isolated or (usually) concentrated before being submitted to the laboratory.

NOTE: If the isolation or concentration occurs in the laboratory, the procedure is usually considered part of the preparation of the test sample from the laboratory sample (in-laboratory processing).

2.4 SAMPLE PREPARATION

2.4.1 In-laboratory processing

The selection, removal, and preparation of the test (or analytical) portions from the laboratory sample.

NOTE: The processing may include a reduction in the size of the unit(s) (division) and in particle size (reduction), as well as mixing to achieve homogeneity (see 2.4.2).

2.4.2 Mixing

The combining of components, particles, or layers into a more homogeneous state.

NOTES: (1) The mixing may be achieved manually or mechanically by shifting the material with stirrers or pumps or by revolving or shaking the container. The process must not permit segregation of particles of different size or properties.

(2) Homogeneity may be considered to have been achieved in a practical sense when the sampling error of the processed portion is negligible compared to the total error of the measurement system.

2.4.3 Reducing

Decreasing the size of the laboratory sample or individual particles, or both.

NOTE: Division of the size of the laboratory sample may be accomplished manually by coning and quartering (2.4.4) or by riffling (2.4.5) or mechanically by rotary dividers. Reduction of particle size may be accomplished by milling or grinding (2.4.6). Simultaneous division and reduction may also be achieved with mills having stream diverters.

2.4.4 Coning and quartering

The reduction in size of a granular or powdered sample by forming a conical heap which is spread out into a circular, flat cake. The cake is divided radially into quarters and two opposite quarters are combined. The other two quarters are discarded. The process is repeated as many times as necessary to obtain the quantity desired for some final use (e.g., as the laboratory sample or as the test sample).

NOTE: If the process is performed only once, coning and quartering is no more efficient than taking alternate portions and discarding the others.

2.4.5 Riffling

The separation of a free-flowing sample into (usually) equal parts by means of a mechanical device composed of diverter chutes.

2.4.6 Milling/grinding

The mechanical reduction of the particle size of a sample by attrition (friction), impact, or cutting.

NOTES: (1) The required particle size of a sample is related to the size of the test portion and the number of particles required to ensure homogeneity among test portions.
(2) The reduction in particle size may sometimes result in particles of different hardness and density, which produces inhomogeneity during the preparation of the test sample or during the withdrawal of the test portion.

2.5 SAMPLING STAGES (see figure)

2.5.1 Increment (applies to bulk materials and large units)

An individual portion of material collected by a single operation of a sampling device.

NOTES: (1) Increments may be reduced individually or tested either (a) individually or (b) combined with other increments with the resulting composite reduced in size and tested as a single unit.
(2) Increments are created by the sampling operation and are usually taken from parts of a lot separated in time or space.
(3) Increments of a bulk population correspond to units of a packaged population.

2.5.2 Primary sample

The collection of one or more increments or units initially taken from a population.

NOTES: (1) The portions may be either combined (composited or bulked sample) or kept separate (gross sample). If combined and mixed to homogeneity, it is a blended bulk sample.
(2) The term "bulk sample" is commonly used in the sampling literature as the sample formed by combining increments. The term "bulk sample" is ambiguous since it could also mean a sample from a bulk lot and it does not indicate whether the increments or units are kept separate or combined. Such use should be discouraged because less ambiguous alternative terms (composite sample, aggregate sample) are available.
(3) "Lot sample" and "batch sample" have also been used for this concept, but they are self limiting terms.
(4) The use of "primary" in this sense is not meant to imply the necessity for multistage sampling.

2.5.3 Reduced sample

A representative part of the primary (composited or gross) sample obtained by a division and reduction process (2.4.3, 2.4.4, 2.4.5).

NOTE: Typically the mass approximates that of the final laboratory sample.

2.5.4 Subsample

A subsample may be:
(a) a portion of the sample obtained by selection or division;
(b) an individual unit of the lot taken as part of the sample;
(c) the final unit of multistage sampling (2.3.9).

NOTE: The term "subsample" is used either in the sense of a "sample of a sample" or as a synonym for "unit." In practice, the meaning is usually apparent from the context or is defined.

2.5.5 Laboratory sample

The sample or subsample(s) sent to or received by the laboratory.

NOTES: (1) When the laboratory sample is further prepared [reduced] by subdividing, mixing, grinding, or by combinations of these operations, the result is the test sample (2.5.6). When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion (2.5.7) is removed from the test sample for the performance of the test or for analysis.

(2) The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

(3) Several laboratory samples may be prepared and sent to different laboratories or to the same laboratory for different purposes. When sent to the same laboratory, the set is generally considered as a single laboratory sample and is documented as a single sample.

2.5.6 Test sample/analytical sample

The sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis.

2.5.7 Test portion/analytical portion

The quantity of material, of proper size for measurement of the concentration or other property of interest, removed from the test sample.

NOTES: (1) The test portion may be taken from the primary sample or from the laboratory sample directly if no preparation of sample is required (e.g., with liquids), but usually it is taken from the prepared test sample.

(2) A unit or increment of proper homogeneity, size, and fineness, needing no further preparation, may be a test portion.

2.5.8 Test solution/analytical solution

The solution prepared by dissolving, with or without reaction, the test portion in a liquid.

2.5.9 Treated solution

The test solution that has been subjected to reaction or separation procedures prior to measurement of some property.

2.5.10 Aliquot

A known amount of a homogeneous material, assumed to be taken with negligible sampling error. The term is usually applied to fluids.

NOTES: (1) The term "aliquot" is usually used when the fractional part is an exact divisor of the whole; the term "aliquant" has been used when the fractional part is not an exact divisor of the whole (e.g., a 15 mL portion is an aliquant of 100 mL).

(2) When a laboratory sample or a test sample is "aliquoted" or otherwise subdivided, the portions have been called split samples.

* * * * * * *

Acknowledgement

The extensive documentation available from the International Organization for Standardization (ISO) provided the starting point for this document.

INDEX

Aliquant	2.5.10
Aliquot	1.2.4, 1.4, 2.5.10
Analytical portion	1.4, 1.8, 2.5.7
Analytical sample	1.8, 2.5.6
Analytical solution	2.5.8
Batch	2.2.3
Batch sample	2.5.2
Bulk/Bulked sample	1.6, 1.7, 2.5.2
Characteristic	2.1.3
Combined sample	1.7, 2.3.10
Composite sample	1.6, 2.3.9, 2.3.10, 2.5.1, 2.5.2, 2.5.3
Coning and quartering	1.6, 2.4.3, 2.4.4
Consignment	2.2.1
Convenience sample	1.4, 1.6, 2.3.5
Determination	1.2.15
Dividing	2.4.3
Duplicate sample	2.3.7
Final sample	1.6
Grinding	2.4.6
Gross sample	1.3.2, 1.6, 2.5.2
Heterogeneity	2.1.4
Homogeneity	2.1.4
Increment	1.4, 1.5, 1.6, 2.3.9, 2.5.1, 2.5.2
Individual	2.2.4, 2.5.1
In-laboratory processing	2.3.11, 2.4.1
Item	2.2.4, 2.5.1
Laboratory sample	1.3.2, 1.4, 1.6, 1.8, 2.5.5
Lot	2.2.2, 2.2.3
Lot sample	1.6, 2.5.2
Matrix	1.2.16
Milling	2.4.6
Mixing	2.4.2
Modified sample	1.3.2, 2.3.10, 2.3.11
Multistage sample	2.3.9, 2.5.2
Partial sample	1.6
Portion	2.2.4, 2.5.1, 2.5.2
Primary sample	1.5, 2.3.9, 2.5.2, 2.5.3
Quartering	1.6, 2.4.3, 2.4.4
Random sample	2.3.1, 2.3.2
Reduced sample	2.5.3
Reduction	2.4.1, 2.4.3, 2.5.3
Referee sample	2.3.6
Reference sample	2.3.6
Replicate sample	2.3.7
Representative sample	2.3.2
Riffling	1.6, 2.4.5
Rolling	1.6,

Sample 1.2, 2.1.1
Sample unit 1.5
Sampling error 1.2, 2.1.1, 2.1.5
Sampling plan 1.1, 2.1.2
Sampling portions 2.3.9
Sampling stages 2.5
Secondary sample 1.3.2, 2.3.9
Segment 1.5, 1.6, 2.2.5
Selective sample(ing) 1.3.4, 2.3.3
Sequential sample 2.3.8
Specimen 1.2.11, 1.4, 1.5, 2.1.1, 2.2.6
Split sample 2.5.10
Stratified sample 2.3.4
Subsample 1.2.14, 2.3.9, 2.5.4

Tertiary sample 2.3.9
Test portion 1.2.6, 1.3.2, 1.4, 1.8, 2.5.7
Test sample 1.3.2, 1.4, 1.8, 2.5.6
Test solution 1.2.7, 1.8, 2.5.8
Treated solution 1.2.8, 1.8, 2.5.9

Umpire sample 2.3.7, 2.3.6
Unit 1.5, 2.2.4, 2.3.9, 2.5.1