A Survey of the Recent Literature on Sampling for Chemical Analysis
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A Survey of the Recent Literature on Sampling for Chemical Analysis

Byron G. Kratochvil*

Chemistry Department
University of Alberta
Edmonton, Alberta, Canada T6G2G2

John K. Taylor

Center for Analytical Chemistry
National Measurement Laboratory
National Bureau of Standards
Washington, DC 20234

*Guest Worker, Center for Analytical Chemistry
   National Bureau of Standards
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Byron G. Kratochvil
University of Alberta, Edmonton, Alberta, Canada

and

John K. Taylor
National Bureau of Standards

Abstract

Sampling is one of the most important steps in chemical analysis, yet it is often poorly planned and executed. One reason is that key information on sampling is scattered and relatively inaccessible. This article summarizes the more important published articles obtained as the result of a literature search to obtain essential background information for the design of sampling plans and protocols for the National Environmental Specimen Bank. Each reference is briefly described so that its applicability to a specific sampling question can be judged. The compilation consists of 56 references on general aspects of sampling, 9 references on sampling agricultural and food products, 14 references on sampling atmospheres and gases, 18 references on sampling water and waste water, and 18 references on sampling miscellaneous materials.

Key words: Chemical analysis; sampling; sampling atmospheres; sampling food; sampling miscellaneous materials; sampling plan; sampling water.

1. Introduction

Sampling is often the least-considered step in a chemical analysis. Typically, the analyst has very little to do with the actual sampling process. The sample received is assumed to be valid and to merit the analytical work requested, which may be substantial. Too frequently, the analyst is not a specialist in the discipline area of the material and must trust the expertise of the specialist in that field. Usually neither is an expert in sampling theory or practice, although more informed on the latter than the former. Accordingly, optimum sampling practices may not have been followed.

There is a considerable amount of information on sampling available in the technical literature but it is not easy to find. Some of it is embedded in the discipline area of the matrix or in materials science. Much of it is presented incidental to other problems so that careful search is needed to discover it. The statistical aspects of sampling for acceptance testing have been extensively covered, but this literature has not been generally applied to materials investigations.
The references presented in this paper result from a literature search in connection with the National Environmental Specimen Bank (NESB). The NESB must pay careful attention to a wide variety of problems ranging from sample selection, handling, subdivision, homogenization, preservation, to treatment prior to analysis. The search for the available information on such problems was necessarily general since there has been little or no prior research in sample banking. Since no extensive compilations of sampling information were discovered in this search, it seemed appropriate to summarize the more important references to assist others who may need to look more closely at the sampling aspects of their analytical problems.

2. General Comments on Sampling

The general aspects of sampling for chemical analysis were reviewed in a recent article by the present authors [See Ref. 37, Section 4A]. The sample and the measurement data share equal importance in almost every chemical compositional investigation. Sometimes the test sample itself is the only item of compositional concern, such as in the case of the identification of a particular specimen for forensic purposes or for the diagnosis of a health problem of a particular individual. More frequently, the test specimen is analyzed because it is considered to be representative, and can provide information about the larger population from which it was drawn. Other problems may be quite complex, involving the measurement of samples collected over intervals of time and space. All situations require careful consideration of sample handling and treatment; and homogenization may also be involved. In most cases, all aspects of sampling must be carefully planned to permit statistical inferences to be drawn from the data.

The following is a brief summary of the most important questions that must be addressed in designing a sampling plan:

* Is the sample unique or a member of a larger population?
* Is the sample considered to be homogeneous or heterogeneous relative to the data requirements? If heterogeneous, is the heterogeneity general, localized, or stratified?
* Is the composition constant or variable with respect to time, temperature, pressure, or other physical conditions?
* What mode of sampling, should be used: random; systematic; continuous; intermittent; discrete; composite?
* Is generally available sampling equipment suitable or are there special requirements with respect to design, materials of construction, and mode of operation? Does the problem require discriminatory or non-discriminatory sampling equipment such as special filters, or iso-kinetic sampling, for example?
* Does the problem require mean compositional data, information about the extremes, or population distribution?

* Will the sample change due to interactions of components, chemical or physical reactions with the container, or due to environmental influences during transit or storage? Is stabilization required? What are the stabilizers that should be used?

* What pre-treatment will be required, such as drying, mixing, pulverization, sterilization, sieving, compositing, removal of "foreign" substances, homogenization, and sub-sampling? What are the conditions and equipment requirements to handle such operations effectively without damage to the sample?

* What number and what size of individual samples are required to yield statistically meaningful results? What are the sampling and measurement sequences required to identify the sample variability in the presence of overall measurement variability?

* What are the quality assurance measures that need to be taken, including the proper identification of samples and an operational chain of custody?

The details of sampling for routine analytical testing, for monitoring industrial processes, for acceptance of materials, and for regulatory compliance are generally well defined and have been standardized in some cases. A number of examples of such are included in Section 4. However, there are many materials investigations for which the sampling plan needs to be individually developed. The references of Section 4 have been annotated to assist in indicating their possible usefulness and/or applicability to a given situation.

3. Search Techniques

The search techniques used in this compilation included a computer search of Chemical Abstracts, using the key words "sampling" and "method", "procedure" or "program", a search of the subject files at the NBS library, and a search of the NBS standards collection (ASTM, ISO, BSI). Because of the vast literature on the sampling of specific substances under specific conditions, and the varying extents to which sampling is treated in conjunction with other areas, it was decided to make the coverage selective. The papers, chapters, and books listed here were selected for inclusion on the basis of their breadth of coverage and range of applicability. In a compilation of this kind the selection necessarily reflects the backgrounds and interests of the authors; we hope that the references included here provide a beginning from which the list of references can be extended in any given area of interest. For this purpose the Science Citation Index is useful.
Much of the pioneering work in sampling has come from research on biological systems, but important contributions have also been made by workers in mining and mineral exploration, and in water, soil, and air studies. Therefore, many fields have contributed to the development of sampling theory, and a variety of approaches are available for application to most sampling problems. Considerable experience and common sense is required to select the most appropriate and efficient sampling plan from this variety.

4. Annotated References

A. General References


Mixtures of coarse and fine sand and of sugar and sand were sampled by five procedures—scoop sampling, cone and quartering, table sampler, chute riffler, and spinning riffler. The spinning riffler was judged superior to the other methods because of low maximum sampler error, high sampler efficiency, and low operator bias.


Recommended practice for the preparation of a sampling plan for a specific material.


Recommended practice for calculating how many units of a lot to include in a sample to estimate, with a prescribed precision, the average of some characteristic of the lot.


Presentation of a rule based on statistical evidence by which to judge evidence based on samples. The rule provides an estimate of the result expected if the entire lot were investigated in the same way as the sample.

An outline of principles to be observed in establishing procedures for sampling several classes of industrial chemicals. Included is information on statistical considerations, equipment, time and place of sampling, sample reduction, and slurry sampling.


A list of definitions for the terms accuracy, assignable cause, lot, universe (population), precision, repeatability, reproducibility, and sample.


A summary of a plenary discussion held at the International Conference: Modern Trends in Activation Analysis, September 1976.


An excellent discussion of sampling by attributes and sampling by variables, including how to calculate the number of samples to analyze in order to achieve a preselected degree of confidence in the results.


A nonmathematical discussion of the statistical analysis of three sampling experiments on chrome are described by Bennett, Eardley, and Beech in reference [11], below.


Treats theory of sampling, computation of sample size, and multiple sampling operations in approximately the first half of the article, and the practice of sampling, including particle size reduction, mixing, and sampling techniques, in the second half. Somewhat out of date, but provides useful background information.
Studies on three chromite ores showed that, for two of them, the composition of the ore was fairly independent of the diameter of the pieces selected in the sample. The third ore was uniform also except for the finer (<1/8 inch diameter) and the largest pieces (12 inches or more in diameter).

A review of the problems of physically obtaining samples of material occurring in bulk form, and of the use of statistics in providing information on the minimum sampling adequate to make a reliable judgment about a lot.

A general discussion of the problems associated with the sampling of chemicals and how to solve them.

A discussion primarily of techniques and methods of sampling fluids, compact solids, and particulate solids, but including definitions and problems of sampling, determination of sample size, and sampling to determine the average of some property of a lot or to identify and measure the variations in a non-homogeneous material.

Background to subsequent parts 2 to 4, which provide specific instruction for sampling gases, liquids, and solids. Part 1 covers definitions, basic purposes and principles of sampling, statistical information, and safety.

A mathematical treatment of the effect of the degree of blending of a composite sample on the adequacy of the sample as a representative of the lot.

A book emphasizing sociological and business surveys, but containing some general information useful to chemical sampling situations. Pages 9-17 treat the role of sampling theory, probability sampling, use of the Gaussian distribution, bias and its effects, and mean square error. Pages 374-389 cover a mathematical model for errors of measurement, effects of constant bias (systematic error), effects of errors that are uncorrected within a sample, effects of intrasample correlation between errors, and treatment of interpenetrating subsamples.


A clear, readable treatment of bulk sampling that emphasizes the need to create a "sampling model" that can be used to determine the sample size needed for each type of bulk material. Included is statistical theory applicable to the sampling of bulk materials.


Quantitative effects of grain size of a rock or mineral sample, and of the composition of contaminating minerals, are evaluated.


The importance of homogeneity in sampling powders for analysis is discussed in terms of the size of a segregation unit relative to the size of the sample. Both theoretical and experimental treatments are included. Much of this work is included in reference [21].


A discussion of the effect of sampling on the determination of trace elements. The Wilson-modified form of the Benedetti-Pichler equation for calculating sampling error is discussed and tested experimentally on two particulate systems. Agreement between the predicted and experimentally-found error was satisfactory.

This book provides an in-depth study of the sources of error in sampling of bulk materials. It is not addressed to users of sampling, but rather to those studying the mechanisms whereby sampling errors are generated.


Sampling plans for evaluating the quantity of the mineral kyanite and for assessing variability at four sampling stages are presented. The minimum sample size required, and the results of a composite sample, are also treated.


A note outlining briefly but clearly how to calculate the sample size required to estimate a population average with some desired precision.


A treatment of the use of average, standard deviation, and variance as statistical descriptors of particulate mixtures is discussed. The precision of the results was found to depend on sampling techniques, number of samples, and nature of the mixture.


The application of statistical methods developed in social survey sampling to the sampling of particulate mixtures for analysis is discussed. The coefficient of correlation provides a measure of the homogeneity of the elements within a sample; the relationship between the coefficient of correlation and sample size is derived for several mixture models.


A note discussing the question of how large a sample must be taken for a single analysis when the component of interest is in discrete small particles.

Excellent discussion of Gaussian distribution, probability, and the binomial theorem in clear, simple language.


A discussion of the problems of preparation of a geological field sample for chemical analysis, and of the use of sampling constants as a guide to the description of the optimum size and the characteristics of samples and subsamples.


Sampling error can be estimated and controlled during analysis of mixtures through use of sampling diagrams based on statistical models that combine Gaussian and Poisson statistics.


Figure 2 in this article illustrates a sampling diagram for the determination of potassium in a biotite, and shows how the sampling error decreases with increasing sample size.


A review of sampling of uniform and segregated materials for chemical analysis.


The major sources of variance in skewed data, large- and small-scale inhomogeneity and subsampling error, are modeled as a double Poisson distribution, the parameters of which reflect the characteristics of the ore-body (relevant variance) and of the sample reduction process (irrelevant variance).
To distinguish between analytical and subsampling error, the use of a sampling constant, defined as $K_s = R^2_w$, where $R$ is the relative standard deviation of a set of measurements of the sought-for substance and $w$ is the weight of material taken for each measurement, is recommended. The approach is considered reliable if the value of $K_s$ is based on ten or more measurements, and if the sought-for material is not present at trace levels in a highly heterogeneous form.

This document covers: (1) general background, including definitions and overall sampling procedures; (2) methods of determining the size and number of increments and forming subsamples, and sampling from moving belts, from bins such as ship holds, and from trucks or railway cars; (3) methods of sample preparation, such as crushing and division, prior to analysis; and the method for determination of particle size distribution of a sample. Appendices describe experimental methods for checking precision or bias in sampling operations, and for evaluating variations in two-stage sampling and in stratified and periodic systematic sampling.

This paper describes how to reach a required accuracy in the composition of a gross sample relative to the lot from which it has been taken. (See Ref. 46 for Part I.)

A general discussion of the importance of sampling, and the development of sampling plans.

An application of the approach of Benedetti-Pichler to the estimation of minimum sample size as a function of particle size.


An equation is derived for the variance of the composition of samples drawn from a mixture of two or more components. Among the factors incorporated is a coefficient of correlation that gives the degree of correlation between the composition of neighboring particles in the mixture.

The properties of the coefficient of correlation described in Part I are examined theoretically and experimentally, and an equation relating the variance between samples and the sample size is derived.

A general discussion of the place of statistical sampling schemes in the field of analytical chemistry, both historically and for the future. The design of a sampling scheme depends almost entirely on the purpose of the analytical operation.

A statistical procedure is outlined for the determination of the effect of sample size, sub-sample size, and particle size on the precision of an assay. In the procedure it is assumed that the active agent is present in only a portion of the particles comprising the sample.

A manual that provides mathematical procedures for determining the optimum size of a research experiment. Emphasis is on engineering applications. Major topics include estimation problems, tests of hypotheses, selection problems, sequential sampling, and lot acceptance sampling.

This publication provides sampling procedures and reference tables for use in planning and conducting inspection by attributes.


A set of sampling plans based on variables rather than on attributes. Plans based on variables are more desirable when measurement data are available because fewer samples need be measured to provide the same level of confidence as for plans based on attributes.


The relation between lot size, sample size, and number of samples is derived for lots with internal correlation. (See Ref. 36 for Part II.)


Sampling procedures are discussed in a general way, with special reference to sampling tools, the applicability of random sampling, and statistical considerations.


This report describes the development of a plan to comprehensively survey the occurrence of potentially toxic substances in a defined geographical area. It describes the basic philosophy of such a survey, the development of the sampling plan, and methods of data reduction using unique computer-generated plots to show concentration profiles.

This paper examines the problems of obtaining representative samples for the analysis of trace elements that are distributed heterogeneously, and for establishing the topographical distribution of these traces.


A discussion of biological sampling and statistics. The first part describes distribution patterns of organisms in nature and the design of monitoring programs. The second part describes distribution models, sampling statistics (including sample size; random, stratified random, and two-stage sampling), and biological diversity.


A treatment of sampling in three parts: general principles, practical problems, and methods of calculating sampling precision. Provides useful general information on the number and size of sample increments. Well written and readable.


A general theory is presented for sampling, based on the premise that the variance in a bulk lot of a material can be expressed by a relation incorporating two sampling constants. The relation may be used to design sampling programs, and to determine in advance the precision of a sampling experiment as a function of sample size and the number of increments. See also discussions of this article listed below:


In this note it is shown that the empirical results of Visman can in part be derived from statistical theory.

In this note Visman and Duncan discuss the generality of the Visman approach, as well as its validity under certain circumstances. Lerner summarizes their points of view, and concludes that the Visman procedure, while not strictly 'general', is a giant step above the previous rules-of-thumb often used.

Visman, J.; Duncan, A. J. Discussion 3 on "A general theory of sampling". J. Mat. 7:345; 1971.

Here Visman develops further the factors that hold the constant relating to segregation variance at a stable value. Duncan agrees with Visman's treatment and conclusions, and develops the statistical theory for Visman's approach.


A readable discussion of the problem of sampling, with many examples from real life, particularly from social and financial sources.


The problems of efficient and economical sampling are discussed under eight points. The authors stress that preliminary information on the standard deviation of sampling is necessary before a rational sampling procedure can be established, or the degree of representativeness of the samples taken be determined.


The sampling errors in a heterogeneous rock powder depend fundamentally on the weight multinominal distribution of the mineral particles. For samples of varying particle size, the total number of particles is of no special significance and should be replaced by a weighted reciprocal mean.
Sampling variance in bulk materials is considered. A library of sampling experience on a given material is shown to be a useful guide to how many samples are required from a given lot to achieve a desired certainty in the result.

B. Sampling Agricultural and Food Products


Excellent overall discussion of sampling problems in foods, presented in a non-theoretical way. Includes examples of sampling for pesticide residues, aflatoxins, and N-nitrosamines.


A brief outline of the problems encountered by the food scientist when removing samples for nutritional analysis from large lots.


A detailed review of the methods developed for the sampling and testing for aflatoxins in foods and feeds. Aflatoxins are the only food contaminants routinely monitored on an international scale at the 10 ppb level.


A discussion of the uncertainty introduced into results for the measurement of aflatoxins in peanuts owing to sampling variance.


The variation associated with various sampling programs was tested. Random sampling was indicated to be cheaper and more accurate than composite sampling.

General conditions relating to the sampling for the assessment of quality, stressing acceptance testing.


General principles are set forth for sampling meat and meat products. Part II in preparation deals with the number of primary samples to be taken from a lot.


General conditions relating to sampling of oil seeds for assessment of quality when purchased as industrial raw products. Equipment and sampling schemes are described. Companion standard ISO 664 establishes a procedure to obtain representative samples.


This is a companion standard to be used with ISO 1573 which describes a drying procedure and method for determination of moisture content.

C. Sampling the Atmosphere and Gases


Seven pages of definitions.


A presentation of the broad concepts of sampling the atmosphere, with emphasis on statistical planning, meteorological factors and effects of topography.

Detailed discussion of apparatus and procedures for obtaining valid samples of gaseous materials for industrial and environmental applications.


Specific details of apparatus and procedure are described in 21 pages.


A useful broad treatment of materials considered as pollutants, especially at trace levels.


A critical evaluation of the apparatus and techniques for collection of spot, continuous, intermittent, and composite samples. Provides a thorough, clear, treatment.


A brief treatment of the apparatus and general approach required to sample organic substances present in air.


A bibliography, including abstracts, of 202 references covering techniques of sampling particles in the earth's atmosphere.


Most recent of a series of biennial reviews on the title topic that includes some references on sampling.

A detailed review with 421 references, of which one-half page and 24 references are devoted to overall sample collection techniques, and an additional one and a half pages and 34 references devoted to adsorption traps, filtration, impingers and impactors, and cryogenic traps for collection of organic compounds.


A detailed treatment of an often neglected component of atmospheres.


A general discussion emphasizing the problem of ensuring that the sample is representative.


A good treatment of the precautions necessary for the collection of a valid sample from a stack.


Analytical results for polycyclic aromatic hydrocarbons are low owing to losses during sampling. The losses depend on the vapor pressure of the individual hydrocarbon, the air flow rate through the sampler, the presence of oxidants such as sulfur dioxide, and the time of sampling.
D. Sampling Water and Waste Water


Sampling errors reported to be much greater than the error in analysis by infrared measurements. A calculation showed that an increase in the number of replicates was generally better than larger samples.


A broadly useful discussion of the apparatus and procedures for obtaining valid samples from streams, lakes, and other bodies of water.


A comprehensive review of the methods of collection, storage, and preservation of water samples for trace metal analysis.


The procedure involves collection in 10-m deep pits with clear plexiglass tubes by operators wearing cleanroom clothes, particle masks, and clean PVC gloves.


Includes sections on sampling and sample handling.


A review of the title topics. About 10 of the 57 references deal with sampling.
This part gives general principles to be applied in the design of sampling programs for quality control and for identification of sources of pollution of water, including bottom sediments. It contains sections on definitions of objectives, identification of sampling situations, time and frequency of sampling, and flow measurements.

Subsequent parts currently being drafted include Part 2: General guidelines to sampling techniques, and Part 3: General recommendations for the preservation and handling of samples.


Provides guidelines for sampling surface waters, with principles of sampling and recommended procedures for sample storage and preservation.


A network of sampling stations is designed on the basis of probability sampling.


A review with four references.


The sampling, storage, and shipping conditions studied had little effect on the total organic carbon levels in tap and river water samples at moderate concentrations, but contamination is possible at low levels.


A discussion emphasizing the problems of sampling, sample handling, and sample transportation.

Too low a frequency of sampling intervals led to large errors in estimation of the phosphorus inputs.


A report treating the mechanics of poly-disperse systems as they affect the collection of analytical samples.


A practical discussion of the major considerations in sampling waste streams. Sample handling and preservation are covered, and equipment available for sampling is described.


A review of the problems and techniques of water sampling. Applicability and performance of individual, composite, and automatic sampling are discussed, as is sample storage.


Discussion includes municipal and industrial waste water as well as water for municipal use.

E. Sampling Miscellaneous Materials


These guidelines include sections on the minimum requirements of a valid sampling program, and a discussion of how to employ statistics in planning and interpreting sampling operations in the environment.

A review of sampling non-ferrous scrap that is rejected by the electrical and electronics industry but contains precious metals. No references are given.


Detailed outline of the procedure necessary for the valid sampling of coal.


The determination of eight different quantities, such as pH, loss on ignition, total nitrogen, exchangeable potassium, and so on was performed on a large number of samples collected by nine different sampling programs. The average results for all the parameters except loss on ignition were found to be statistically significantly different between programs.


Objectives, models of ecological systems, frequency distributions and sampling designs are discussed. No universal model appears applicable at present; instead, various individual models serve specific purposes. The use of body size (length and weight) as an auxiliary variable in sampling is recommended for either comparison or estimation.


A list of three requirements for an acceptable physiological sample is given: appropriate physical state of patient, such as fasting; blood samples that are as representative as possible; and proper storage and preservation of the sample.


A report describing the tests conducted to determine the characteristics of stockpiled munitions.

Six 24-hour urine specimens must be collected from a child for sodium measurement if the probability of misclassifying a child in the top third relative to the bottom third is to be held to 0.01. The ratio of intra- to inter-individual variances was estimated to be 1.94 for 24-hour urine sodium.


A critical survey of the title topic, with emphasis on procedures that are most likely to maintain high sample integrity.


A procedure for testing the microhomogeneity of such standard reference materials as alloys and glasses is described. An electron microprobe is programmed to take measurements on the sample each 1 to 10 μm along the surface.


A general discussion, with emphasis on the importance of representative sampling. Few details of sampling operations are provided.


Tourniquets may induce hemoconcentrations, leading to high levels of serum protein and protein-bound substances. Increases of 5 to 13 percent were observed. Calcium and magnesium were similarly affected.

A review with eight references on the factors affecting segregation in liquid alloys upon solidification. Includes a discussion of milling and drilling techniques for the sampling of solid alloys, and ways of preparing homogeneous samples from molten alloys.


A review with many references about sampling and sample preparation of human body tissues and fluids.


Sampling errors in ion microprobe analysis of heterogeneous materials are investigated. Use of the sampling constant concept is suggested. Equations that allow estimation of the degree of heterogeneity in a sample are presented, and procedures to determine the number of replicate analyses necessary to achieve a desired precision are outlined.


A discussion emphasizing the need to minimize contamination when sampling and storing clinical materials. Blood collection is used as an example.


A review discussing the collection, preparation, and analysis of vegetation and soils for inorganic residues of gaseous and particulate air pollutants. Includes 140 references.


Description of a sampling program, "Variables Sampling Plan", is given. The program is designed to maintain fuel quality with minimum costs in analysis and fuel production.
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National Standard Reference Data Series—Provides quantitative data on the physical and chemical properties of materials, compiled from the world’s literature and critically evaluated. Developed under a worldwide program coordinated by NBS under the authority of the National Standard Data Act (Public Law 90-396).

NOTE: The principal publication outlet for the foregoing data is the Journal of Physical and Chemical Reference Data (JPCRD) published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements available from ACS, 1155 Sixteenth St., NW, Washington, DC 20036.

Building Science Series—Disseminates technical information developed at the Bureau on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

Technical Notes—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NBS under the sponsorship of other government agencies.

Voluntary Product Standards—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The standards establish nationally recognized requirements for products, and provide all concerned interests with a basis for common understanding of the characteristics of the products. NBS administers this program as a supplement to the activities of the private sector standardizing organizations.

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