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NATIONAL BUREAU OF STANDARDS

NBS TECHNICAL NOTE 1153

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

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

NBS technical note

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Issued January 1982

National Bureau of Standards Technical Note 1153

Nat. Bur. Stand. (U.S.), Tech. Note 1153, 27 pages (Jan. 1982)
CODEN: NBTNAE

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 1982

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A Survey of the Recent Literature on Sampling for Chemical Analysis

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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

[1] Allen, T.; Khan, A. A. Critical evaluation of powder sampling procedures. Chem. Eng. 238:108-112; 1970.

> 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.

[2] American Society for Testing and Materials, Probability sampling of materials, ANSI/ASTM E105-58 (Reapproved 1975) Philadelphia, 1975.

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

[3] American Society for Testing and Materials, Choice of sample size to estimate the average quality of a lot or process, ANSI/ASTM E122-72 (Reapproved 1979) Philadelphia, 1979.

> 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.

[4] American Society for Testing and Materials, Acceptance of evidence based on the results of probability sampling, ANSI/ASTM E141-69 (Reapproved 1975) Philadelphia, 1975.

> 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.

[5] American Society for Testing and Materials, Sampling industrial chemicals. ANSI/ASTM E300-73 (Reapproved 1979) Philadelphia, 1979.

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.

[6] American Society for Testing Materials, Definitions of terms relating to statistical methods. ANSI/ASTM E456-72, Philadelphia, 1972.

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

[7] Anders, O. U. Representative sampling and the proper use of reference materials. Anal. Chem. 49:33A-36A; 1977.

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

[8] Bauer, E. L. A statistical manual for chemists, 2nd Ed. New York: Academic Press; 1971. 131-144.

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.

[9] Beech, D. G. Sampling of hard heterogeneous materials. II. Detailed statistical treatment. Trans. Brit. Ceram. Soc. 70:13-17; 1971.

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

[10] Benedetti-Pichler, A. A. Theory and principles of sampling for chemical analysis, chapter in Physical methods in chemical analysis, Vol. III. W. G. Berl, ed. New York: Academic Press; 1956. 183-217.

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.

[11] Bennett, H.; Eardley, R. P.; Beech, D. G. Sampling of hard heterogeneous materials. I. Derivation of a new and simple technique. Trans. Brit. Ceram. Soc. 70:1-12; 1971.

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).

[12] Bicking, C. A. The sampling of bulk materials. Mat. Res. Stds. 7:95-116; 1967.

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.

[13] Bicking, C. A. Sampling, Kirk-Othmer Encyclopedia of Chemical Technology, 2nd Ed. 17:744-762; 1968.

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

[14] Bicking, C. A. Principles and methods of sampling, chapter in Treatise on analytical chemistry, 2nd Ed., Vol. I. I. M. Kolthoff and P. J. Elving, eds. New York: John Wiley & Sons; 1979. 299-359.

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 nonhomogeneous material.

[15] British Standards Institution. Methods for sampling chemical products. Part 1. Introduction and general principles. BS 5309, London; 1976.

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.

[16] Brown, G. H.; Fisher, N. I. Subsampling a mixture of sampled material. Technometrics 14:663-668; 1972.

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.

[17] Cochran, W. G. Sampling techniques, 2nd Ed., New York: John Wiley & Sons; 1963. 9-17 and 374-389.

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.

[18] Duncan, A. J. Bulk sampling, Section 25A, in Quality control handbook. J. M. Juran, ed. New York: McGraw-Hill; 1974. 25A1-25A13.

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.

[19] Engels, J. C.; Ingamells, C. O. Effect of sample inhomogeneity on K-Ar dating. Geochim. Cosmochim. Acta 34:1007-1017; 1970.

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

[20] Grant, C. L.; Pelton, P. A. Role of homogeneity in powder sampling, ASTM Special Technical Publication No. 540; American Society for Testing and Materials. Philadelphia, 1973, 16-29.

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].

[21] Grant, C. L.; Pelton, P. A. Influence of sampling on the quality of analyses with emphasis on powders. Advances in X-ray Analysis 17:44-67; 1974.

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.

[22] Gy, P. Theory and practice of sampling bulk particulate mixtures. New York: Elsevier; 1979.

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.

[23] Hackler, W. C.; Clatfeller, T. E.; Farley, J. M.; Heckler, C. L.; Rilee, E. E., Jr. Statistics in sampling of bulk material. Ceramic Bull. 52:882; 1973.

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.

[24] Hahn, G. J. How large a sample do I need for 95% confidence? CHEMTECH 5:61-62; 1975.

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

[25] Harnby, N. The statistical analysis of particulate mixtures. Part 1. The sampling of mixtures and the resultant precision of estimates based on the sample. Powder Technology 5:81-86; 1971/72.

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.

[26] Harnby, N. The statistical analysis of particulate mixtures. Part 2. The application of social survey statistical techniques to solids mixing problems. Powder Technology 5:155-165; 1971/72.

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.

[27] Harris, W. E.; Kratochvil, B. Sampling variance in analysis for trace components in solids. Anal. Chem. 46:313-315; 1974.

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.

[28] Heiland, R. E.; Richardson, W. J. Work sampling. New York: McGraw-Hill; 1957.

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

[29] Ingamells, C. O. New approaches to geochemical analysis and sampling. Talanta 21:141-155; 1974.

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.

[30] Ingamells, C. O. Control of geochemical error through sampling and subsampling diagrams. Geochem. Cosmochim. Acta 38:1225-1237; 1974.

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.

[31] Ingamells, C. O. Preparation, analysis, and sampling constants for a biotite, in Accuracy in trace analysis. Nat. Bur. Stand. (U.S.) Spec. Publ. 422; 1976 August. p 415.

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.

[32] Ingamells, C. O. General sampling theory, in Sampling and assaying precious metals. Corrigan, D. A.; Browning, B., eds. Proceedings of second international seminar; 1980; Brooklyn, New York, Int. Precious Met. Inst., Inc., pp 39-63.

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

[33] Ingamells, C. O.; Pitard, F. F.; Fox, J. J.; Samland, P. K.; Bright, M. J. Evaluation of skewed exploration data. Extended abstracts - international symposium anal. chem. explor. min. process mater. CSIR, Pretoria, South Africa; 1977. pp 208-13.

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).

[34] Ingamells, C. O.; Switzer, P. A proposed sampling constant for use in geochemical analysis. Talanta 20:547-68; 1973.

To distinguish between analytical and subsampling error, the use of a sampling constant, defined as $\underline{K_s} = \underline{R}^2\underline{w}$, where $\underline{R}$ is the relative standard deviation of a set of measurements of the sought-for substance and $\underline{w}$ is the weight of material taken for each measurement, is recommended. The approach is considered reliable if the value of $\underline{K_s}$ is based on ten or more measurements, and if the sought-for material is not present at trace levels in a highly heterogeneous form.

[35] Japanese Industrial Standard, General rules for methods of sampling bulk materials, JIS M 1800-1973, Japanese Standards Association. English translation, Tokyo; 1975. 75 pp.

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.

[36] Kateman, G.; Muskens, P. J. W. M. Sampling of internally correlated lots. The reproducibility of gross samples as function of sample size, lot size, and number of samples. II. Implications for practical sampling and analysis. Anal. Chim. Acta 103:11-20; 1978.

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.)

[37] Kratochvil, B. G.; Taylor, J. K. Sampling for chemical analysis. Anal. Chem. 53:A-938A; 1981.

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

[38] Kleeman, A. W. Sampling error in the chemical analysis of rocks. J. Geol. Soc. Aust. 14:43-47; 1967.

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

[39] Kristensen, H. G. Statistical properties of random and non-random mixtures of dry solids. Part I. A general expression for the variance of the composition of samples. Powder Technology 1:249-257; 1973.

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.

[40] Kristensen, H. G. Statistical properties of random and non-random mixtures of dry solids. Part II. Variance-sample size relationships derived by autocorrelation theories. Powder Technology 8:149-157; 1973.

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.

[41] Ku, H. H. Statistical sampling and environmental trace organic analysis, in Trace organic analysis: A new frontier in analytical chemistry. Nat. Bur. Stand. (U.S.) Spec. Publ. 519; 1979 April. pp 1-6.

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.

[42] Kwolek, W. F.; Lillehoj, E. B. Analytical variation when proportions of sampled units contain the active agent. J. Assoc. Off. Anal. Chem. 59:787-794; 1976.

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.

[43] Mace, A. E. Sample size determination. New York: Reinhold; 1964.

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.

[44] Military Standard 105D, Sampling procedures and tables for inspection by attributes. Washington, D.C.: U. S. Government Printing Office; 1963 April.

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

[45] Military Standard 414, Sampling procedures and tables for inspection by variables for percent defective. Washington, D.C.: U. S. Government Printing Office; 1957.

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.

[46] Muskens, P. J. W. M.; Kateman, G. Sampling of internally correlated lots. The reproducibility of gross samples as a function of sample size, lot size, and number of samples. Part I. Theory. Anal. Chem. Acta 103:1-9; 1978.

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

[47] Quackenbush, F. W.; Rund, R. C. The continuing problem of sampling. J. Assoc. Off. Anal. Chemists 50:997-1006; 1967.

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

[48] Scheide, E. P.; Filliben, J. J.; Taylor, J. K. Survey of the occurrence of mercury, lead, and cadmium in the Washington, D.C. area. NBSIR 78-1428; 1977.

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.

[49] Skogerboe, R. K.; Morrison, G. H. Role of sampling in trace analysis. In Preprints of contributed papers. Symposium on trace characterization-chemical and physical. Nat. Bur. Stand (U.S.). 1966 October. pp 589-595.

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.

[50] Slack, K. V.; Averett, R. C.; Greeson, P. E.; Lipscomb, R. G. Methods for collection and analysis of aquatic biological and microbiological samples. Chapter A4 of Book 5, Laboratory analysis in techniques of water-resources investigations of the U. S. Geological Survey. Washington, D.C.: U. S. Government Printing Office; 1973. 6-26.

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.

[51] Tomlinson, R. C. Sampling, in Comprehensive analytical chemistry, Vol. IA. W. L. Wilson; D. W. Wilson, eds. New York: Elsevier; 1959. 36-75.

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.

[52] Visman, J. A general sampling theory. Mat. Res. Stds. 9:9; 1969.

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:

- Duncan, A. J. Comments on "A general sampling theory", Mat. Res. Stds. 11(1):25; 1971.

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

- Visman, J.; Duncan, A. J.; Lerner, M. Further discussion: "A general theory of sampling", Mat. Res. Stds. 11(8):32; 1971.

  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.

[53] Williams, W. H. A sampler on sampling. New York: Wiley; 1978.

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

[54] Wilrich, P. Th.; Leers, K. J. Statistical considerations in the sampling of bulk goods. Ber. Dt. Keram. Ges. 51:266; 1974.

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.

[55] Wilson, A. D. The sampling of silicate rock powders for chemical analysis. Analyst 89:18; 1964.

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.

[56] Youden, W. J. The role of statistics in regulatory work. J. Assoc. Off. Analyt. Chemists 50:1011-1013; 1967.

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

[1] Horwitz, W.; Howard, J. W. Sampling methods in trace organic analysis in food. Nat. Bur. Stand (U.S.) Spec. Publ. 519; 1979 April. pp 231-242.

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.

[2] Lento, H. G. The role of sample preparation in nutritional food labeling. Nat. Bur. Stand. (U.S.) Spec. Publ. 519; 1979 April. pp 243-248.

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

[3] Schuller, D. L.; Horwitz, W.; Stoloff, L. Review of sampling plans and collaboratively studied methods of analysis for aflatoxin. J. Assoc. Off. Anal. Chem. 59:1315-1343; 1976.

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.

[4] Whitaker, T. B.; Dickens, J. W. Variability of aflatoxin test results. J. Am. Oil Chem. Soc. 51:214-218; 1974.

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

[5] Williams, C. J.; Peterson, R. G. Variation in estimates of milk fat, protein, and lactose content associated with various bulk milk sampling programs. J. Dairy Sci. 61:1093-1102; 1978.

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

[6] International Standards Organization, Fresh fruits and vegetables - Sampling. ISO 874; 1980.

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

[7] International Standards Organization, Meat and meat products - Part I - Taking primary samples. ISO 3100/1; 1975.

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.

[8] International Standards Organization, Oilseeds - Sampling. ISO 874; 1980.

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.

[9] International Standards Organization, Tea - Preparation of ground samples of known moisture content. ISO 1572; 1975.

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

[1] American Society for Testing and Materials. Standard definitions of terms relating to atmospheric sampling and analysis. ANSI/ASTM D1356; 1973.

Seven pages of definitions.

[2] American Society for Testing and Materials. Standard recommended practice for planning the sampling of the atmosphere. ANSI Z257.1/ ASTM D1357; 1957.

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

[3] American Society for Testing and Materials. Standard recommended practice for sampling atmospheres for analysis of gases and vapors. ANSI Z257.2/ASTM D1605; 1960.

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

[4] American Society for Testing and Materials. Standard method for sampling stacks for particulate matter. ANSI Z257.3/ASTM D2928; 1971.

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

[5] Axelrod, H. D.; Lodge, J. P. Sampling and calibration of gaseous pollutants, chapter in Air pollution, 3rd ed., Vol. 3. A. C. Stone, ed.; 1976. 146-182.

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

[6] British Standards Institution. Methods for sampling chemical products. Part 2. Sampling of gases. BS 5309, Part 2; 1976. 21 p.

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

[7] Bunn, W. W.; Deane, E. R.; Klein, D. W.; Kleopfer, R. D. Sampling and characterization of air for organic compounds. Water, Air, Soil Pollution. 4:367; 1975.

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

[8] Carvagnaro, D. M. Air pollution sampling of particles. National Technical Information Service PB80-802853; 1979. 209 p.

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

[9] Fox, D. L.; Jefferies, H. E. Air pollution. Anal. Chem. 51:22R-; 1979.

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

[10] Lamb, S. I.; Petrowski, C.; Kaplan, I. R.; Simoneit, B. R. T. Organic compounds in the urban atmosphere: a review of distribution, collection, and analysis. J. Air Pollut. Control Assoc. 30:1098-1115; 1980.

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

[11] Liu, B. Y. H., ed., Fine particles: Aerosol generation, measurement, sampling, and analysis. New York: Academic Press; 1976. 837 p.

A detailed treatment of an often neglected component of atmospheres.

[12] Lodge, J. P., Jr. Accuracy in air sampling. Nat. Bur. Stand. (U.S.) Spec. Publ. 422; 1976 August. pp 311-320.

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

[13] Morrow, N. L.; Brief, R. S.; Bertrand, R. R. Sampling and analyzing air pollution sources. Chem. Eng. 24:84-98; 1972 January 24.

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

[14] Tomingas, R. Remarks on the sampling procedures for polycyclic aromatic hydrocarbons from the atmosphere. Fresenius' Z. Anal. Chem. 297:97-101; 1979.

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

[1] Ahmed, S. M.; Beasley, M. D.; Efromson, A. C.; Hites, R. A. Sampling errors in the quantitation of petroleum in Boston Harbor water. Anal. Chem. 46:1858-1860; 1974.

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.

[2] American Society for Testing and Materials. Standard practice for sampling water. ASTM D3370; 1976.

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

[3] Batley, G. E.; Gardner, D. Sampling and storage of natural waters for trace metal analysis. Water Res. 11:745-756; 1977.

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

[4] Bowtron, C. Reduction of contamination problems in sampling of antarctic snows for trace element analysis. Anal. Chim. Acta 106:127; 1979.

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

[5] Grasshoff, K., ed., Methods of seawater analysis. Weinheim: Verlag Chemie; 1976.

Includes sections on sampling and sample handling.

[6] Hensley, C. P.; Jeffer, W. J.; McKenzie, C.; Lair, M. D. Continuous monitoring, automated analysis, and sampling procedures. J. Water Pollut. Control Fed. 50:1061-1066; 1978.

A review of the title topics. About 10 of the 57 references deal with sampling.

[7] International Standards Organization, Water quality - Sampling - Part 1; Guidance on the design of sampling programmes. ISO 5667/1-1980(E); 27 p.

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.

[8] Kingsford, M. Sampling of surface waters. Water, Soil Tech. Publ. 2:15; 1977.

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

[9] Liebetrau, A. M. Water quality sampling: some statistical considerations. Water Resour. Res. 15:1717-1725; 1979.

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

[10] Miller, J. M. The collection and chemical analysis of precipitation. Proc. nat. conf. complete water reuse, 2nd; 1975; Pub. 1976; 331-337.

A review with four references.

[11] Otson, R.; Williams, D. T.; Bothwell, P. D.; McCullough, R. S.; Tate, R. A. Effects of sampling, shipping, and storage on total organic carbon levels in water samples. Bull. Environ. Contam. Toxicol. 23:311-318; 1979.

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.

[12] Ottendorfer, L. J. Analytical problems concerning natural waters. Proc. anal. div. chem. soc. 15:52; 1978.

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

[13] Sharpley, A. N. Phosphorus inputs into a stream draining on agricultural watershed. Water, Air, Soil Pollution 6:39; 1976.

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

[14] Shelley, P. E. Sampling of water and wastewater. National Technical Information Service, PB Rep., PB-272664; 1977.

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

[15] Staff of Research and Education Association, Sampling in waste-water monitoring, chapter 33 in Modern pollution control technology, Vol. II. New York: Research and Education Association; 1978. 33-1 to 33-25.

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

[16] Thompson, R. J. Sampling and analysis techniques in current use in the EPA/NOAA/WMO precipitation network. World Meteorological Organization (Publ.) 460:40; 1977.

[17] Wagner, R. Sampling and sample preparation. Water, Z. Anal. Chem. 282:315-321; 1976.

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.

[18] Ziegler, W. J. Variability in water analysis introduced by sample collection, preservation, or preparation techniques. Proc. ann. ind. air water pollut./contam. control semin. 9:14; 1979.

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

E. Sampling Miscellaneous Materials

[1] ACS Subcommittee on Environmental Chemistry. Guidelines for data acquisition and data quality evaluation in environmental chemistry. Anal. Chem. 52:2242-2249; 1980.

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.

[2] Allard, M. Sampling and sample preparation of scrap. Erzmetall 32:437-440; 1979.

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

[3] American Pulp and Paper Institute. Standard for sampling and analysis of coal. TAPPI T6050S.

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

[4] Ball, D. F.; Williams, W. F. Variability of soil chemical properties; efficiency of sampling programs on an uncultivated brown earth. J. Soil Science 22:60-68; 1971.

The determination of eight different quantities, such as pH, loss on ignition, total nitrogen, exchangable 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.

[5] Eberhardt, L. L.; Gilbert, R. O.; Hollister, H. L.; Thomas, J. M. Sampling for contaminants in ecological systems. Environ. Sci. Technol. 10:917-925; 1976.

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.

[6] Ibbott, F. A. Sampling for clinical chemistry. Nat. Bur. Stand. (U.S.) Spec. Publ. 422; 1976 August. pp 353-360.

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.

[7] Lawhorne, S. Demilitarization plan: operation of the chemical agent munitions disposal system (CAMDS) at Tooele Army Depot, inclosure number 2, statistically significant sampling program. National Technical Information Service, DRCPM-DRD-TR-74017; Report 1977; 21 p.

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

[8] Liu, K.; Cooper, R.; Soltero, I.; Stamler, J. Variability in 24-hour urine sodium excretion in children. Hypertension (Dallas) 1:631-636; 1979.

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.

[9] Maienthal, E. J.; Becker, D. A. A survey of current literature on sampling, sample handling, and long term storage for environmental materials. Nat. Bur. Stand. (U.S.) Tech Note 929; 1976 October. 40 p.

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

[10] Marinenko, R. B.; Heinrich, K. F. J.; Ruegg, F. C. Microhomogeneity of NBS standards. Proc. Ann. Conf. - Microbeam Anal. Soc., 14th, 1979; 221-226.

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.

[11] Maxwell, J. A. Sampling and sample preparation at the geological survey of Canada. Nat. Bur. Stand. (U.S.) Spec. Pub. 422; 1976 August. pp 285-297.

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

[12] McNair, P.; Nielsen, S. L.; Christiansen, C.; Axelsson, C. Gross errors made by routine blood sampling from two sites using a tourniquet applied at different positions. Clin. Chim. Acta 98:113-118; 1979.

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.

[13] Protzer, H. Sampling of alloys susceptible to segregation. Erzmetall 32:430-437; 1979.

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.

[14] Sansoni, B.; Iyengar, V. Sampling and sample preparation methods for the analysis of trace elements in biological material. Spez. Ber. Kernforschangsanlage Juelich. Juel-Spez 13; 1978; 79 p.

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

[15] Scilla, G. J.; Morrison, G. H. Sampling error in ion microprobe analysis. Anal. Chem. 49:1529-1536; 1977.

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.

[16] Spucke, A.; Hoste, J.; Versieck, J. Sampling of biological materials. Nat. Bur. Stand. (U.S.) Spec. Publ. 422; 1976 August. pp 299-310.

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

[17] Temple, P. J.; Wills, R. Sampling and analysis of plants and soils. Methodol. Assess. Air Pollut. Eff. Veg., Handb. Spec. Conf. 1978, Paper No. 13, Heck, W. W.; Krupa, S. V.; Linzon, S. N., eds. Pittsburgh: APCA; 1979. 23 p.

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

[18] Zeigler, R. K.; Matlack, G. M.; Rein, J. E.; Metz, C. F. Statistically designed program for sampling and chemical analysis of reactor fuel materials. National Technical Information Service, LA-DC-12812; Report 1971; 15 p.

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.

NBS-114A (REV. 2-8C)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET (See instructions)	1. PUBLICATION OR REPORT NO. NBS TN 1153	2. Performing Organ. Report No.	3. Publication Date January 1982

4. TITLE AND SUBTITLE

A Survey of the Recent Literature on Sampling for Chemical Analysis

5. AUTHOR(S)

Byron G. Kratochvil and John K. Taylor

6. PERFORMING ORGANIZATION (If joint or other than NBS, see instructions)

NATIONAL BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
WASHINGTON, D.C. 20234

7. Contract/Grant No.

8. Type of Report & Period Covered

Final

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP)

Same as above

10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

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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.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

Chemical analysis; sampling; sampling atmospheres; sampling food; sampling miscellaneous materials; sampling plan; sampling water.

13. AVAILABILITY

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27

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