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TERMINOLOGY IN SOIL SAMPLING

(IUPAC Recommendations 2005)

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Terminology in soil sampling

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Abstract: The scientist must be sure that, within a stated context, the terms used in articles, publications, or the daily conversation among colleagues are intended by all in the same precise way, without any possible ambiguity. As already pointed out by "Nomenclature for sampling in analytical chemistry (IUPAC Recommendations 1990)" [4], it is not acceptable that scientists are not able to orient themselves in a sampling or analytical process. This can occur if the terms used are not well defined. Moreover, to better appreciate the development of new theories or concepts, progressive updates can be necessary. To this end, on the basis of the existing terminology documents and of the most recent knowledge in the field of soil sampling, an updated terminology in sampling (specifically, soil sampling) is recommended.

Keywords: soil; sampling; recommendations; analytical chemistry; terminology, Division V.

CONTENTS

1. INTRODUCTION

Recently, there have been a growing number of efforts to overcome confusion, ambiguity, and contradictions in the usage of terms and clarification of their definitions in the field of sampling. The IUPAC Recommendations, "Nomenclature for sampling in analytical chemistry", published in 1990 [4], and the ISO standard 11074-2, "Soil Quality – Vocabulary – Part 2, 1998" [1], are the most widely used terminology documents related to soil sampling. However, recent developments and studies of various sampling aspects (i.e., uncertainty quantification, method validation, comparison of sampling tools, and strategies) require new concepts to be developed and also some new terms to be introduced for their description.

One of the outcomes of the SOILSAMP international project, funded and coordinated by the Italian Environmental Protection Agency (APAT, Italy) and aimed at assessing the uncertainty associated with soil sampling in agricultural, semi-natural, urban, and contaminated environments, was an updated terminology in sampling.

This document is the result of that effort, and is intended to present terms and definitions to be used in the field of soil sampling and sampling uncertainty. A set of geostatistical terms, of interest in the context of soil sampling and sampling uncertainty estimation, is also illustrated together with the recommended definitions.

2. SOIL SAMPLING TERMINOLOGY

A wide variety of terms are used in the practice of sampling [6]. However, many terms identify a specific (single) operation, and, in some cases, different terms are used to describe the same concept. Furthermore, it is quite usual in the scientific literature that different authors use different terms to describe the same operation or concept. Also, in works produced by standardization bodies, it is possible to find different terms for describing the same operation or concept as well as a different philosophy of organizing the concepts themselves.

In this paper, the selection of terms recommended for use in connection with soil sampling is made with the aim to improve the consistency of related sampling terminology. In addition, some new terms are proposed. To facilitate the understanding and real meaning of such terms, schemes illustrating the relationship of the operations related to sampling are presented (see Figs. 1 and 2).

Sampling (Fig. 1), following the indication of a sampling plan, starts from the selection of the potentially impacted "sampling sites". Samples are then collected at the different "sampling points" accurately located. The number and the relative position of the "sampling points" depend on the scope of sampling and thus on the particular "sampling pattern/strategy", which can be selected on a statistical basis.

"Increments" are taken from each "sampling point" to produce a "primary sample". When mixed together, a "composite/aggregate sample" is obtained.

A "laboratory sample" is obtained either directly from the "primary sample" or by "reducing" the "primary sample" or the "composite samples". During this phase, "coning and quartering", "quartering", "riffling", "grinding", etc. may be necessary. The laboratory sample is then packed and shipped to the laboratory for required pretreatment and characterization (chemical, physical, biological, etc.).

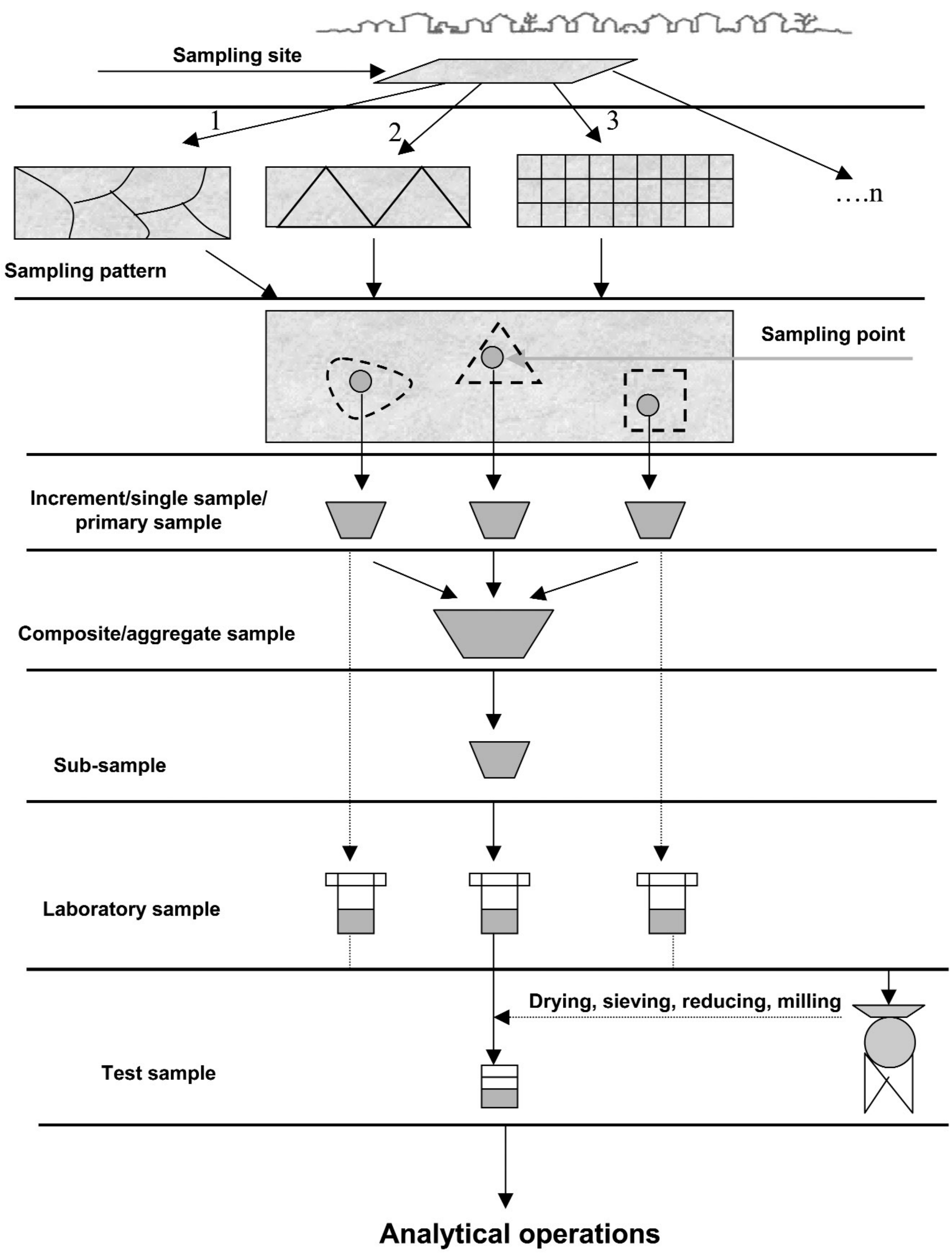

Fig. 1 Relationships of sampling operations.

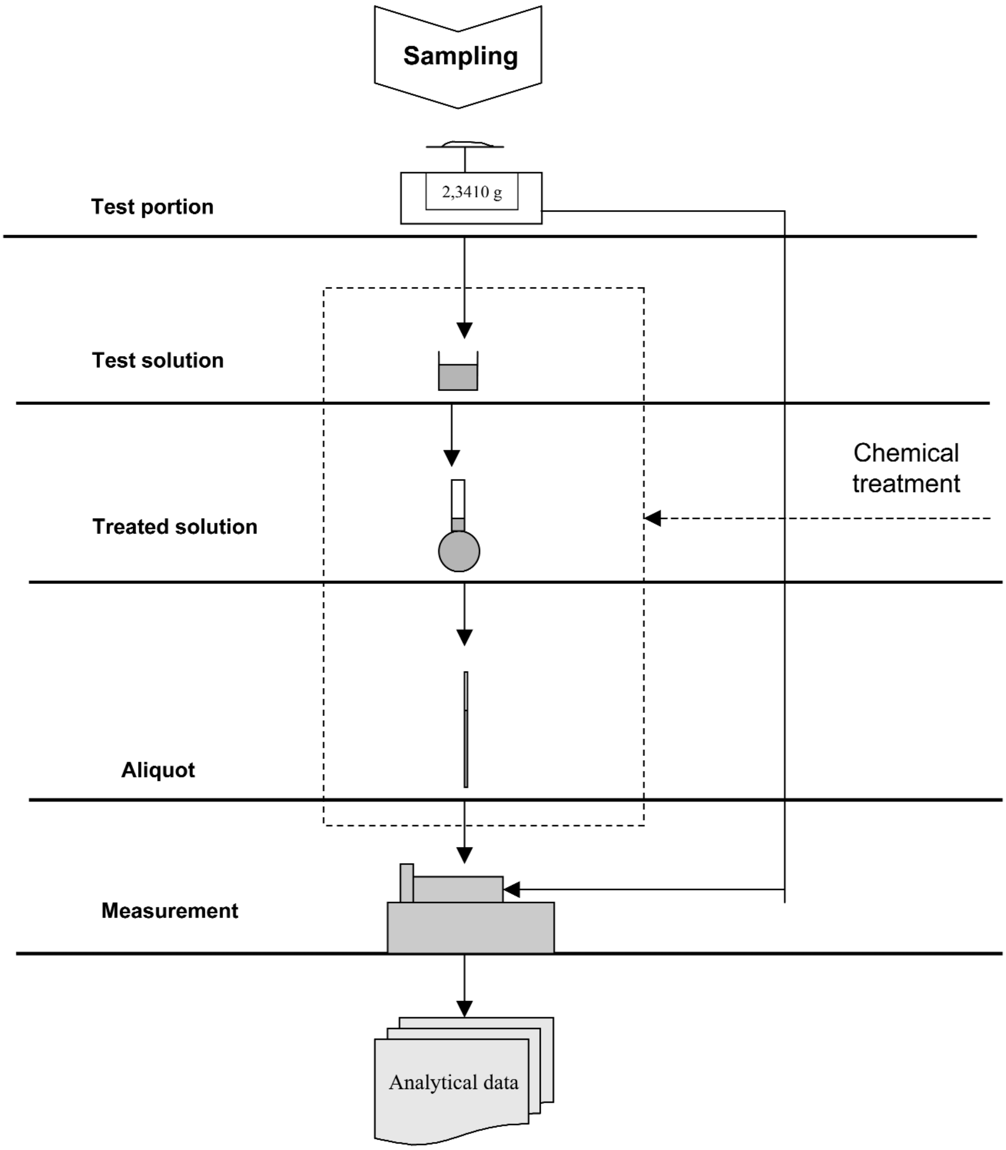

Fig. 2 Relationships of analytical operations.

2.1 Terms and definitions

Composite sample (average sample, aggregate sample) (ISO 11074-2, 1998) [1]: "Two or more increments or sub-samples mixed together in appropriate proportions, either discretely or continuously (blended composite sample), from which the average value of a desired characteristic may be obtained"[1];

[1]IUPAC, 1990 considers the composite sample only with reference to sampling of bulk material when more increments from the bulk are combined to obtain a physically averaged sample. However, the term "composite sample" is not specified.

Increment (IUPAC, 1990) [4]: "Individual portion of material collected by a single operation of a sampling device"[2];

Informative judgmental sampling: "Sampling in which locations are chosen according to the judgement of an expert and partly in accordance with the statistical principles of sampling"[3];

Judgmental sampling (ISO 11074-2, 1998) [1]: "Sampling in which locations are chosen according to the judgement of an expert";

Laboratory sample (IUPAC, 1990) [4]: "The sample or sub-sample sent to or received by the laboratory"[4];

Parent population (ISO 11074-2, 1998) [1]: "Totality of items under consideration";

Primary sample (IUPAC, 1990) [4]: "The collection of one or more increments or units initially taken from a population";

Replicate (duplicate) samples (ISO 11074-2, 1998) [1]: "One of the two or more samples or sub-samples obtained separately at the same time by the same sampling procedure or sub-sampling procedure"[5];

Representative sample: "Sample resulting from a sampling plan that can be expected to reflect adequately the properties of interest in the parent population";

Sample (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "A portion of material selected from a larger quantity of material";

Sample size (ISO 11074-2, 1998) [1]: "Number of items or the quantity of material constituting a sample";

Sampler: "Person or group of persons carrying out the sampling procedures at the sampling point"[6];

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[2]Increments are usually combined with other increments, with the resulting composite reduced in size and tested as a single unit. This concept is in agreement with the definition proposed by ISO 11074-2, 1998 that intends the increment only being used in a composite sample. In the case of unit to be kept separate, the ISO standard uses the term "single sample". IUPAC, 1990 foresees the situation in which an increment can be analyzed separately.

[3]The judgment of an expert should be based on a prior knowledge of the sampling site and/or on the visual site observation. It allows the selection of the sampling pattern/strategy, the number of the samples being collected, and, in general, the location of the sampling points.

[4]Even if the definitions in ISO 11074-2 [1] and IUPAC, 1990 [4] are slightly different, the notes attached are the same. Mainly, it is noted that the laboratory sample is the final sample from the point of view of the sample collection, but it is the initial sample from the point of view of the laboratory.

[5]In IUPAC 1990 [4], it is stressed that the comparability has to be assured in space and/or time. The Note is the same in both standards. Although the replicate samples are expected to be identical, often the only thing replicated is the act of taking the physical sample. In the case of soil sampling, the concept of replicate can be associated to the concept of sampling points as is intended the act of taking two or more samples close to each other.

[6]It is based on the original definition by ISO 11074-2, 1998, in which the term "sampling locality", instead of "sampling point", is used, even if not previously defined. Tools and other devices to obtain samples are sometimes also designated "samplers". In this case, the terms "sampling devices" or "sampling equipment" should be used. The sampler should have specific knowledge and experience in soil sampling.

Sampling (ISO 11074-2, 1998) [1]: "Process of obtaining a sample"[7];

Sampling design (ISO 11074-2, 1998) [1]: "Arrangement by which a sampling programme is to be conducted"[8];

Sampling device (ISO 11074-2, 1998) [1]: "Apparatus/tool to obtain a sample";

Sampling pattern/sampling strategy: "The result of the selection of the sampling points within a sampling site"[9];

Sampling plan (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Predetermined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a population as a sample";

Sampling point: "The place where sampling occurs within the sampling site"[10];

Sampling procedures (ISO 11074-2, 1998) [1]: "Operational requirements and/or instructions relating to the use of a particular sampling plan";

Sampling site: "A well delimited area, where sampling operations take place"[11];

Sampling techniques (ISO 11074-2, 1998) [1] : "All appropriate procedures and sampling devices used to obtain and describe samples of soil, either in the field or during transportation and in the laboratory";

Simple random sample (ISO 11074-2, 1998) [1]: "Sample of n items taken from a population of N items in such a way that all possible combinations of n items have the same probability of being taken";

Stratification (ISO 3534-1, 1993) [2]: "The division of a population into mutually exclusive and exhaustive sub-populations (called strata), which are thought to be more homogeneous with respect to the characteristics investigated than the total population";

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[7]Sampling, ordinarily ends, after pretreatment steps, with the removal of the test (or analytical) portion from the test (or analytical) sample. For "test portion" and "test sample", see the related definitions (Section 3.1). "Sampling" also relates to the selection of locations for the purpose of in situ testing carried out in the field without removal of material.

[8]The purpose of designing a sampling program is to provide the most efficient methods to reach valid and relevant conclusions from the investigations of soil, with due regard to cost or resource use commensurate with sampling program objectives. The design is a function of many considerations such as the aim of the investigation, the (degree of) heterogeneity of the material under consideration, and the cost of performing the investigation.

[9]Sampling patterns are selected (using or not a statistical approach) according to the scope of sampling and to the need of obtaining representative samples.

[10]In ISO 11074-2, 1998 [1], this term is associated with the precise identification of the position where the sampling operation occurs. A general sampling point can be accurately located, and the accuracy will depend on the surveying method used. If the need to vary the position of the sampling point during the sampling exercise is expected, it could be convenient to carry out an accurate location once the sampling exercise is completed rather than before it starts or as it progresses. On the other side, in soil sampling for agricultural investigation, if composite samples have to be collected, a precise identification of the sampling points may be not crucial. In same cases, the location of a sampling point on the ground is an important requirement (i.e., modeling studies, subsequent development of geographic information system, etc.).

[11]For example, within the frame of a contaminated soil surveys, the borders of the site should be selected in order to include all the possible zones that may be reached by the contamination. In the first instance, the borders are usually administrative borders or factory perimeters.

Single sample (ISO 11074-2, 1998) [1]: "Sampling unit collected by a single operation of a sampling device and kept and treated separately from others";

Stratified sample (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Sample obtained from strata or subparts, putatively homogeneous of the parent population"[12];

Stratified sampling (ISO 3534-1, 1993) [2]: "In a population which can be divided into mutually exclusive and exhaustive sub-populations (called strata), sampling carried out in such a way that specified proportions of the sample are drawn from the different strata and each stratum is sampled with at least one sampling unit";

Sub-sample (ISO 11074-2, 1998) [1]: "Sample obtained by a procedure in which the items of interest are randomly distributed in parts of equal or unequal size"[13];

Sub-sampling (sample division) (ISO 11074-2, 1998) [1]: "Process of selection of one or more sub-samples from a sample of a population"[14];

Systematic sampling (ISO 11074-2, 1998) [1]: "Sampling by some logical and organized method";

Unit, item, portion, individual (IUPAC, 1990; ISO 11074-2, 1998)[15] [1,4]: "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, tested or combined".

3. OPERATIONS RELATED TO SAMPLE PRETREATMENT AND ANALYSIS

Pretreatment can be performed either at the sampling site in order to obtain the "laboratory sample" or at the laboratory in order to obtain a "test sample" from the "laboratory sample" for analytical purposes.

At the laboratory, the "laboratory sample" is split (by "reducing", "milling", etc.) into a number of "test samples" to be used for the analytical characterization.

According to the particular analytical test, a "test portion" is obtained from the "test sample" (Fig. 2) and submitted to the various analytical steps. For example, if dissolution/extraction and clean-up operations are required, an aliquot of the treated test solution is finally sent to the measurement. If no treatment is foreseen, the test portion (solid) is directly measured.

3.1 Terms and definitions

Aliquot (ISO 11074-2, 1998) [1]: "Known amount of a homogeneous material, assumed to be taken with negligible sampling error";

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[12]The objective of taking stratified samples is to obtain a more representative sample than that which might otherwise be obtained by random sampling. A mean value of a measurand for the whole lot may be determined on a blended composite sample from individual increments or mathematically obtained from separate determinations on each increment.

[13]The Note in ISO 11074-2, 1998 [1] corresponds totally to the definition of "sub-sample" in IUPAC, 1990. That is "A sub-sample may be: a) a portion of the sampled obtained by selection and division; b) an individual unit of the lot taken as part of the sample; c) the final unit of the multistage sampling" (ISO 11074-2, def. 3.6 [1]).

[14]In IUPAC, 1990 [4], the term "sub-sampling" does not exist, but at point 2.4 (Sample preparation) are reported the definitions related to different procedures for the sample division such as "reducing", "coning and quartering", and "riffling". Some of these definitions are in compliance.

[15]The term "unit" can be also used to identify the portion of soil surface at the "sampling site" in which the "sampling point" is located.

Comminution: "Decreasing the size of individual particles within a sample"[16];

Coning and quartering (IUPAC, 1990) [4]: "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 time as necessary to obtain the quantity desired for some final use (e.g., as the laboratory sample or as the test sample)"

Homogeneity/heterogeneity (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Degree to which a property or a constituent is uniformly distributed throughout a quantity of material";

Milling, grinding (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Mechanical reduction of the particle size of a sample by attrition, impact or cutting";

Mixing (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Combining of components, particles or layers into a more homogeneous state";

Quartering: "The reduction in size into quarters of a granular or powdered sample. Two opposite quarters are combined, while other two quarters are discarded. The process is repeated as many time as necessary to obtain the quantity desired for some final use (e.g., as the laboratory sample or as the test sample)";

Reducing (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Decreasing the size of the laboratory sample or individual particles, or both"[17];

Riffling (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Separation of a free-flowing sample into (usually) equal parts by means of a mechanical device composed of diverter chutes";

Sample pretreatment (ISO 11074-2, 1998) [1]: "Collective noun for all procedures used for conditioning a soil sample to a definite state which allows subsequent examination or analysis or long-term storage";

Test portion (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Quantity of material, of proper size for measurement of the concentration or other property of interest, removed from the test sample"[18];

Test sample (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Sample, prepared from the laboratory sample, from which the test portions are removed for testing or for analysis".

4. SAMPLING UNCERTAINTY TERMINOLOGY

All measurements are affected by uncertainty. This reflects the property of a measurement to determine a range of values that can be reasonably attributable to the quantity subject of the measurement. Many sources could influence the overall measurement uncertainty, and, in general, all of them necessarily should be identified and, if possible, quantified.

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[16]This term should be used if it is required to separate the process of reducing the particle size of a sample from that of reducing the sample mass.

[17]If the reduction operation is aimed only at changing the particle size of the sample, the term "comminution" could be appropriate.

[18]The portion may be either combined or kept separate. If combined and mixed to homogeneity, it is a blended bulk sample.

Although scientists have, in general, already accepted these concepts, and a lot of effort has been made by standardization bodies to develop guides and rules, there is still confusion on colloquial usage of terms related to uncertainty. For example, terms such as "error" are normally used instead of "uncertainty", contributing in some cases to possible misunderstanding, and the precise limits of application of the terms are not always well understood. In general, the error can be detected but not quantified, as the true value of a measurement can never be determined. On the contrary, uncertainty is a measurable parameter, being based on the evaluation of the statistical distribution of the results of a series of measurements, described in general by standard deviations and variances.

In the frame of soil sampling and analysis, the uncertainty and all related terms have to be the basis of a common language. If in the analytical field a framework of the uncertainty terminology has been defined, the same effort on sampling has not yet produced a consensus. Sampling affects the analytical results, as well as sample preparation and treatment, contributing to the total measurement uncertainty. Sampling uncertainty can be properly quantified, following different approaches and considering different situations (matrices, environment, parameter, concentration). Sampling uncertainty can be assumed as a parameter of the quality of sampling, in order to compare different sampling strategies/devices, to assess the sampling performance, and, finally, to select an appropriate sampling technique and protocol for stated objectives.

Tools and parameters normally related to the analytical field and QA/QC schemes, such as reference materials, can find some analogs in the sampling field. From this point of view, terms such as "reference sampling" and "reference site" need to be explained. Through a reference sampling on a selected area, which is an intensive sampling performed by a single operator/sampler using a single sample device and following a defined pattern and protocol and the subsequent analysis of the samples collected, a well-characterized reference site can be obtained. Therefore, the quantity of a soil property (i.e., trace element concentration or pedo-chemical parameters, etc.) in each possible sample location within the reference site can be well known with its uncertainty. The reference site is nothing more than a natural matrix reference material for sampling. When not all elements of interest are naturally present in this reference site, but one or more selected elements (of suitable, known, quantity, and concentration) are added and homogenized into the soil, a synthetic reference material for sampling is obtained.

4.1 Terms and definitions

Accuracy (ISO, 1993) [2]: "The closeness of the agreement between the result of a measurement and a true value of the measurand";

Bias (ISO 3534-1, 1993) [2]: "The difference between the expectation of the test results and an accepted reference value"[19];

Characteristic (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "Property or attribute of a material that is measured, compared and noted";

Error (of measurement) (ISO, 1993) [2]: "The result of a measurement minus a true value of the measurand";

Measurand (ISO, 1993) [2]: "Particular quantity subject to measurement";

[19]Bias is the total systematic error contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value.

Measurement (ISO, 1993) [2]: "Set of operations having the object of determining the value of a quantity"[20];

Precision (ISO 3534-1, 1993; ISO, 1993) [2]: "The closeness of the agreement between independent test results obtained under stipulated conditions";

Reference sampling: "Characterization of an area, using a single sampling device and a single laboratory, to a detail allowing the set-up of a distribution model in order to predict, with a known uncertainty, element concentrations at any sampling point" [21];

Reference site: "Area, one or more of whose element concentrations are well characterised in terms of spatial and temporal variability" [22];

Sampling error (IUPAC, 1990; ISO 11074-2, 1998) [1,4]: "The part of the total error (the estimate from a sample minus the population value) associated with only a fraction of the population and extrapolated to the whole, as distinct from analytical or test error";

Sampling uncertainty: "The part of the total measurement uncertainty attributable to sampling";

True value (ISO, 1993) [2]: "Value consistent with the definition of a given particular quantity".

Uncertainty (of measurement) (ISO, 1993) [2]: "A parameter associated with the result of a measurement that characterizes the dispersion of the values that could be reasonably attributed to the measurand";

5. GEOSTATISTICAL TERMINOLOGY

Many of the elaborations made by classical statistics are based on a root assumption: the data must be independent. The real situation, particularly in the soil environment, is that this is not true. A state of non-independence is the most common condition between soil samples and the effect of spatial (or temporal) fluctuation and variability has to be accounted for. In the recent past, geostatistics has been demonstrated to be a useful system for estimation of concentration of elements in soil at unsampled locations. At the same time, it represents a powerful tool suitable for tackling problems associated with models describing the spatial distribution of elements in the soil [5]. This technique enables the data (and the collected samples), taking into account the correlation existing between different soil samples in space (or in time), to be assessed. In Fig. 3, the flow chart of a hypothetical geostatistic process to be applied to analytical data is illustrated. A preliminary screening, to detect errors (human or instrumental) and mistakes, is needed to avoid adverse influences on the subsequent spatial analysis. Summary statistics allow the main characteristics of the data and their statistical distribution to be understood. Data transformation could be required in case the data are not normally distributed (commonly, a log-normal distribution characterizes the environmental data). Since data can have some long-range (distance) trend over an area, this must be detected and, eventually, removed. Structural analysis allows an experimental (or sample) variogram to be computed from the data. The experimental variogram reveals valuable information about the spatial correlation structure of the data. The spatial variation may not

[20]The measurement process includes the sampling operation.

[21]The term recalls the chance, after an intensive sampling carried out by a single operator/sampler using a single sample device and following a defined pattern and protocol, to adopt models, i.e., based on interpolation technique, describing the spatial distribution of elements within the reference site.

[22]The definition originates from the ISO-30 [3] "reference material" term.

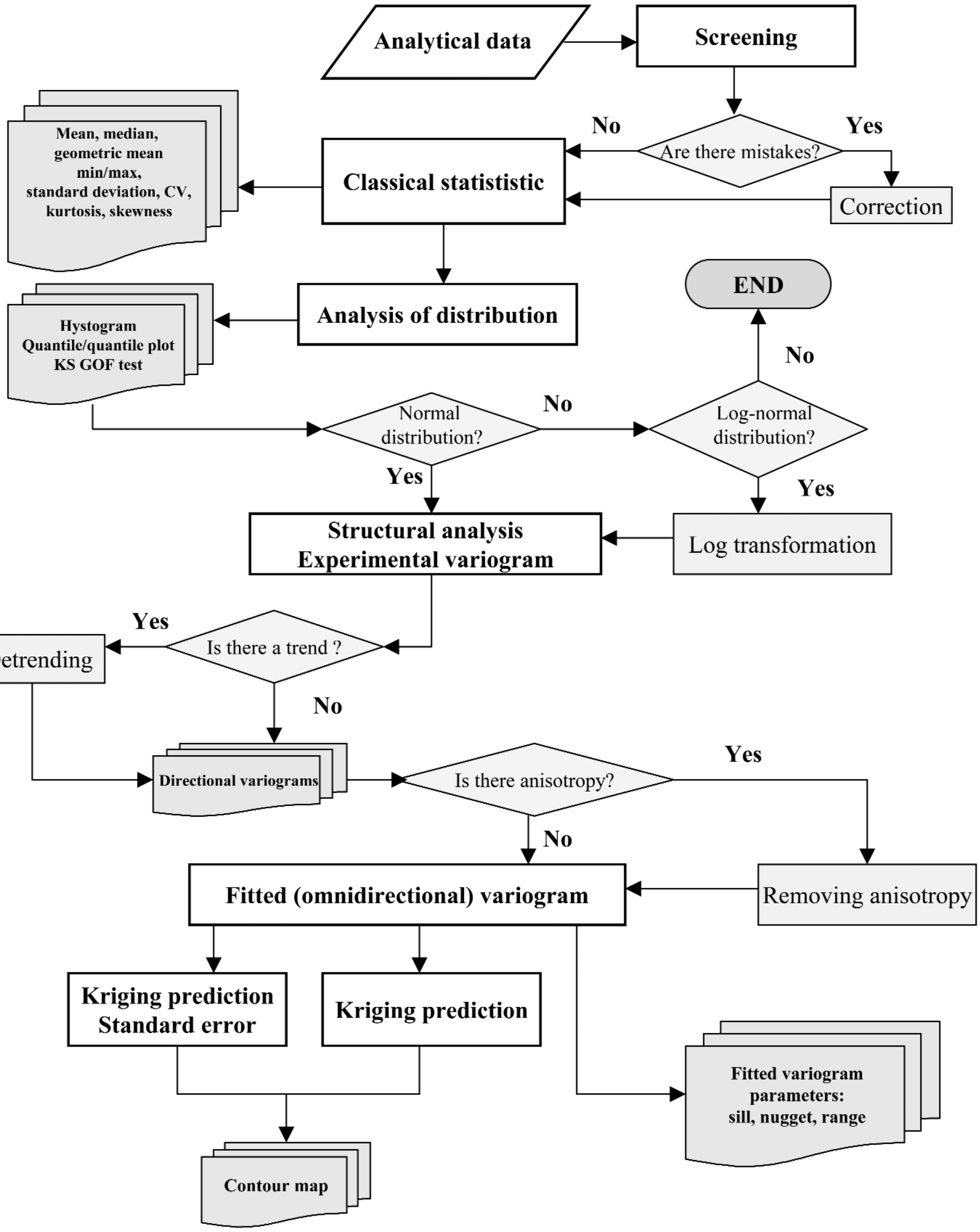

Fig. 3 Geostatistical process.

necessarily be the same in all directions. This so-called anisotropy must be taken into account, before fitting a suitable model for the variogram. The fitted variogram model allows one to interpolate the data, i.e., to predict the values at unsampled locations. The interpolated results can be displayed as isopleth or isoline maps.

Soil sampling uncertainty, as a component of the total measurement uncertainty, can be described and quantified by information obtained through some geostatistical parameters combined with parameters (standard deviation and variance) from classical statistics. Sill, nugget, and range (see Fig. 4) are geostatistical terms that help to account properly for the spatially correlated and uncorrelated components of the variance. The spatially uncorrelated component includes the variance due to analytical operations, sampling, sample preparation/reduction, and other unexplained sources of spatially uncorrelated variance.

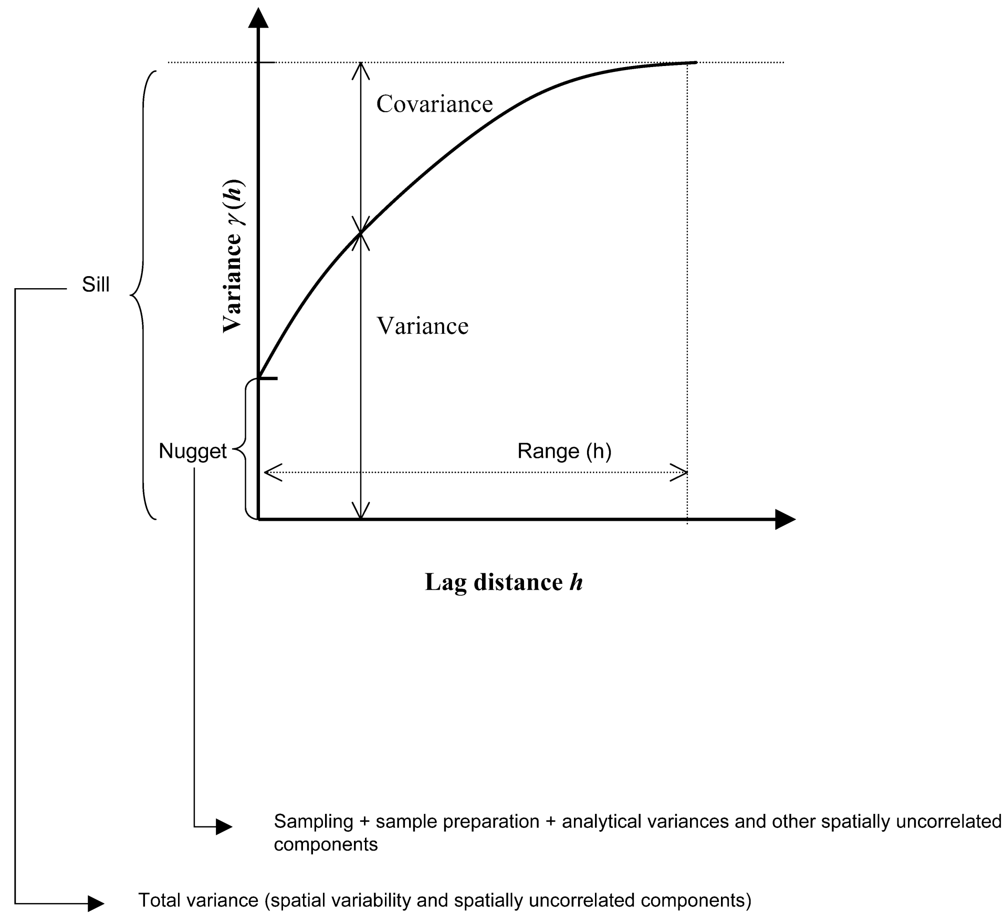

Fig. 4 Variogram and main parameters.

5.1 Terms and definitions

Anisotropy: “A property of a variogram/semivariogram to have different spatial variation structures depending on direction”;

Detrending: “The process of removing a trend”;

Geostatistics: “ Branch of statistics that deals with space/time interpolation of observed data”;

Interpolation/prediction: “The estimation of the values of a variable at unsampled location from observations at surrounding points”;

Kriging: "Suite of interpolation techniques based on regionalized variable theory to predict without bias and minimum variance the value of a variable at unsampled locations "[23];

Lag: "A user-defined distance class within which the semivariance is computed for a set of data points";

Nugget: "The value of the variogram/semivariogram at near zero lag"[24];

Partial sill: "The part of total sill, described in terms of semivariance, minus the nugget"[25];

Range: "A parameter of the variogram/semivariogram representing the distance beyond which the variogram becomes constant";

Regionalized variable theory: "The theory that assumes that the spatial variation of any variable can be expressed as the sum of three components, namely a deterministic function describing the structural component having a constant mean or trend, a stochastic, a random spatially correlated residual from the deterministic function, and spatially uncorrelated random Gaussian noise"[26];

Sill: "The value of the variogram/semivariogram for distance beyond the range of the variogram/semivariogram"[27];

Spatial variation: "The phenomenon that attributes vary in space";

Support: "Physical size of the sample referred to a particular area, depth, and/or volume"[28];

Trend: "Systematic spatial variation of the local mean of variable, usually expressed as a polynomial function of location coordinates"[29];

Variogram/semivariogram: A plot of the semivariance of paired data as a function of the lag, and optionally of the direction, between samples"[30].

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[23]Different methods of Kriging exist, and these are used depending on the objective of the study and the available data. If a variable is correlated with a secondary variable, which is sampled at more locations, the primary variable may be interpolated using co-Kriging. Disjunctive Kriging is the interpolation model that enables estimation of the probability that at a stated point a value of a variable is below or above a threshold value. If the mean value of a variable is to be predicted at a larger support than the sample support, block Kriging is usually applied. Universal Kriging is the interpolation method used in the presence of a trend of the values of the variables observed.

[24]Nugget effect is due to different components of variance, independent to each other, and distinct from the variance linked to spatial variability. Nugget is a variance estimation of the part of the variogram that has no spatial component, comprehensive of variance due to errors in sampling, measurement, short-range spatial variation at distances within the smallest intersample spacing, micro-scale variabilities, and other unexplained spatially uncorrelated sources of variance.

[25]The partial sill represents the variance attributable only to the spatially correlated components.

[26]The definition is extrapolated from the book *Principles of Geographical Information System*, edited by P. A. Burrough and R. A. McDonnell, 1998.

[27]The sill is described as a semivariance that assumes the maximum value at the distance beyond which the variables are considered to be spatially uncorrelated.

[28]The concept of support is similar to unit, if this is characterized in terms of volume. For example, a support can be a unit of stated length and diameter core. Different supports can produce effects on sample distribution and statistics.

[29]The trend is a surface defined by a function of parameters of x- and y-coordinates.

[30]The variogram/semivariogram is mainly described by three parameters: sill, nugget, and range.

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