
**Statistical aspects of sampling from bulk
materials —**

**Part 2:
Sampling of particulate materials**

Aspects statistiques de l'échantillonnage des matériaux en vrac —

Partie 2: Échantillonnage des matériaux particuliers



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 11648 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 11648-2 was prepared by Technical Committee ISO/TC 69, *Applications of statistical methods*, Subcommittee SC 3, *Application of statistical methods in standardization*.

ISO 11648 consists of the following parts, under the general title *Statistical aspects of sampling from bulk materials*:

- *Part 1: General principles*
- *Part 2: Sampling of particulate materials*

It is the intention of ISO/TC 69/SC 3 to develop additional parts to ISO 11648 to cover the sampling of liquids and gases, if the need exists.

Annexes A to J of this part of ISO 11648 are for information only.

Introduction

This part of ISO 11648 gives the basic methods for sampling bulk particulate materials in bulk (e.g. ores, mineral concentrates, coal, industrial chemicals in powder and granular form, and agricultural products such as grain) from moving streams and stationary situations.

Part 1 of ISO 11648 gives a broad outline of the statistical aspects of sampling from bulk materials.

... ..

Statistical aspects of sampling from bulk materials —

Part 2: Sampling of particulate materials

1 Scope

This part of ISO 11648 establishes the basic methods for sampling particulate materials in bulk (e.g. ores, mineral concentrates, coal, industrial chemicals in powder or granular form, and agricultural products such as grain) from moving streams and stationary situations, including stopped-belt sampling, to provide samples for measuring one or more variables in an unbiased manner and with a known degree of precision. The variables are measured by chemical analysis and/or physical testing. These sampling methods are applicable to materials that require inspection to verify compliance with product specifications or contract settlements, to calculate the value of the lot mean of a measurable quantity as a basis for settlement between trading partners, or to estimate the set of variables and variances that describes a system or procedure.

Stopped-belt sampling is the reference method against which other sampling procedures are compared. Dynamic sampling from moving streams is the preferred method whereby a sampling device (called a cutter) is passed through the stream of the particulate material. A complete cross-section of the moving stream can be removed as a primary increment at a conveyor belt transfer point with a falling-stream cutter, or removed from the belt with a cross-belt cutter. In both cases, the selection and extraction of increments can be described by a one-dimensional dynamic sampling model.

Static sampling of bulk material from stationary situations, such as stockpiles, rail or road wagons, the holds of ships and barges, silos, and even comparatively small volumes, is used only where sampling from moving streams is not possible. Such sampling from three-dimensional lots is prone to systematic errors, because some parts of the lot usually have reduced or no chance of being collected for the gross sample. This is in violation of the requirement of the three-dimensional sampling model that all parts have an equal probability of being collected. The procedures described in this part of ISO 11648 for sampling from stationary lots of bulk particulate material with implements such as mechanical augers merely minimize some of the systematic sampling errors.

For these reasons, this part of ISO 11648 is primarily concerned with dynamic sampling from moving streams or stopped-belt static sampling from conveyor belts and is based on a sampling model for one-dimensional lots. Nonetheless, procedures for static sampling from three-dimensional lots are provided where these situations cannot be avoided.

This part of ISO 11648 is concerned with the methods of sampling particulate materials in bulk with the objective of obtaining unbiased measurements of one or more variables of the material with a known degree of precision. However, it does not provide methods for deciding whether to accept or reject a bulk material lot with specified degrees of risk of accepting a sub-standard lot, or of rejecting what is in fact an acceptable lot. These latter procedures are usually called acceptance sampling or sampling inspection methods.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 11648. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 11648 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 3084, *Iron ores — Experimental methods for evaluation of quality variation.*

ISO 3085, *Iron ores — Experimental methods for checking the precision of sampling.*

ISO 3086, *Iron ores — Experimental methods for checking the bias of sampling.*

ISO 3534 (all parts), *Statistics — Vocabulary and symbols.*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method.*

ISO 5725-4, *Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method.*

ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values.*

ISO 11648-1:—¹⁾, *Statistical aspects of sampling from bulk materials — Part 1: General principles.*

ISO 13909-7:—¹⁾, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing.*

ISO 13909-8:—¹⁾, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias.*

Guide to the expression of uncertainty in measurement (GUM). BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML, 1st edition, 1995.

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purpose of this part of ISO 11648, the terms and definitions of ISO 3534 (all parts) and the following (taken from the current draft revision of ISO 3534-2) apply.

NOTE The text <bulk material> shown after terms means the definition given is confined to the field of bulk material sampling.

¹⁾ To be published.

3.1.1**bulk material**

amount of material within which component parts are not initially distinguishable on the macroscopic level

3.1.2**sample**

⟨bulk material⟩ subset of a specified population made up of one or more sampling units

3.1.3**sampling**

act of drawing or constituting a sample

3.1.4**simple random sampling**

sampling where a sample of n sampling units is taken from a population in such a way that all combinations of n sampling units have the same probability of being taken

NOTE In bulk material sampling, if the sampling unit is an increment, the positioning, delimitation and extraction of increments should ensure that all sampling units have an equal probability of being selected.

3.1.5**stratum**

mutually exclusive and exhaustive sub-population considered to be more homogeneous with respect to the characteristic investigated than the total population

EXAMPLES In bulk material, strata, based on time, mass and space, are typically production periods (e.g. 15 min), production masses (e.g. 100 t), holds in vessels, wagons in a train, or containers.

3.1.6**stratified sampling**

sampling such that portions of the sample are drawn from the different strata and each stratum is sampled with at least one sampling unit

NOTE In some cases, the portions are specified proportions determined in advance. However, in post-stratified sampling, the specified proportions would not be known in advance.

3.1.7**stratified simple random sampling**

simple random sampling from each stratum

3.1.8**systematic sampling**

sampling according to a methodical scheme

NOTE 1 In bulk sampling, systematic sampling may be achieved by taking items at fixed distances or after time intervals of fixed length. Intervals may, for example, be on a mass or time basis. In the case of a mass basis, sampling units or increments should be of equal mass. With respect to a time basis, sampling units or increments should be taken from a moving stream or conveyor, for example, at uniform time intervals. In this case, the mass of each sampling unit or increment should be proportional to the mass flow rate at the instant of taking the entity or increment.

NOTE 2 If the lot is divided into strata, stratified systematic sampling can be carried out by taking increments at the same relative locations within each stratum.

3.1.9**sampling unit**

⟨bulk material⟩ one of the member parts, each with equal probability of selection in sampling, into which a population, comprised of the total quantity of bulk material under consideration, is divided

NOTE 1 In bulk sampling, the operative characteristics of the sampling unit are that the probability of selection of all sampling units should be equal and that the entire sampling unit becomes part of the sample when it is selected.

NOTE 2 When sampling from a bulk material is performed by extraction of individual increments, the sampling unit is the primary increment.

3.1.10

precision

closeness of agreement between independent test results obtained under stipulated conditions

NOTE1 Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

NOTE 2 The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test result. Less precision is reflected by a larger standard deviation.

NOTE 3 Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme stipulated conditions.

3.1.11

bias

difference between the expectation of a test result and an accepted reference value

NOTE 1 Bias is the total systematic error as 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.

NOTE 2 The bias of a measurement instrument is normally estimated by averaging the error of indication over an appropriate number of repeated measurements. In this case, the error of indication is the

“indication of a measuring instrument minus a true value of the corresponding input quantity”.

3.1.12

lot

⟨bulk material⟩ definite part of a population, comprised of the total quantity of bulk material under consideration, and where this part is considered as a quantity of material for which specific characteristics are to be determined

NOTE Commerce in bulk material often encompasses transactions involving single lots, and, in these cases, the lot becomes the population.

3.1.13

sub-lot

⟨bulk material⟩ definite part of a lot of bulk material

3.1.14

increment

⟨bulk material⟩ quantity of bulk material taken in one action by a sampling device

NOTE 1 The positioning, delimitation and extraction of the increment should ensure that all parts of the bulk material in the lot have an equal probability of being selected.

NOTE 2 Sampling is often carried out in progressive mechanical stages, in which case it is necessary to distinguish between a primary increment which is a sampling unit that is extracted from the lot at the first sampling stage, and a secondary increment which is extracted from the primary increment at the secondary sampling stage, and so on.

3.1.15

composite sample

⟨bulk material⟩ aggregation of two or more increments taken from a lot

3.1.16

gross sample

⟨bulk material⟩ aggregation of all the increments taken from a sub-lot or lot by the procedures of routine sampling

3.1.17

test sample

⟨bulk material⟩ sample, as prepared for testing or analysis, the whole quantity or a part of it being used for testing or analysis at one time

NOTE The term may be used in such ways as “test sample for chemical analysis”, “test sample for moisture determination”, “test sample for particle size determination” and “test sample for physical testing”.

3.1.18

test portion

⟨bulk material⟩ part of a test sample which is used for analysis or testing at one time

3.1.19

multi-stage sampling

⟨bulk material⟩ sampling in which the sample is selected by stages, the sampling units at each stage being sampled from the larger sampling units chosen at the previous stage

3.1.20

routine sampling

⟨bulk material⟩ sampling for commercial purposes carried out by the stipulated procedures in the specific International Standard in order to determine the average quality of the lot

NOTE The term “regular sampling” is sometimes used as an alternative to “routine sampling”.

3.1.21

experimental sampling

⟨bulk material⟩ non-routine sampling where special purpose experimental design is applied to investigate sources of variance and/or sampling bias

3.1.22

interpenetrating sampling

⟨bulk material⟩ replicate sampling from several lots or sub-lots, where for each lot i or sub-lot i , consecutive primary increments are diverted in rotation into different containers to give multiple composite samples (A_i , B_i , C_i , ...) in order to investigate the variance between the increments in the lot or the sub-lot

NOTE 1 The term “interleaved sampling” is sometimes used as an alternative to “interpenetrating sampling”.

NOTE 2 Most interpenetrating sampling schemes use a duplicate sampling method with composite sample pairs (A_i , B_i) being constituted for each lot i or sub-lot i .

3.1.23

replicate sampling

⟨bulk material⟩ sampling where increments are taken simultaneously or consecutively in pairs in order to constitute multiple composite samples

3.1.24

duplicate sampling

⟨bulk material⟩ replicate sampling where increments are taken simultaneously or consecutively in pairs in order to constitute two composite samples

NOTE Duplicate sampling is a special case of replicate sampling.

3.1.25

manual sampling

⟨bulk material⟩ collection of increments by human effort

3.1.26

mechanical sampling

⟨bulk material⟩ collection of increments by mechanical means

3.1.27

cut

⟨bulk material⟩ single traverse of the sample cutter, in mechanical sampling, through the stream

3.1.28

sample preparation

〈bulk material〉 set of material operations necessary to transform a sample into a test sample

EXAMPLE Reduction of sizes, mixing and dividing.

NOTE For particulate materials, the completion of each operation of sample division defines the commencement of the next sample preparation stage. Thus the number of stages in sample preparation is equal to the number of divisions made.

3.1.29

sample reduction

〈bulk material〉 process in sample preparation whereby the particle size is reduced by crushing, grinding or pulverization

3.1.30

sample division

〈bulk material〉 process in sample preparation whereby a sample of a bulk material is divided by such means as riffling, mechanical division, or quartering into separate parts, one or more of which is retained

EXAMPLE Riffling, mechanical division or quartering.

3.1.31

fixed ratio division

〈bulk material〉 sample division in which the retained parts from individual samples are a constant proportion of the original

3.1.32

fixed mass division

〈bulk material〉 sample division in which the retained divided parts are of almost uniform mass, irrespective of variations in mass of the samples being divided

3.1.33

sample drying

〈bulk material〉 process in sample preparation of partial drying of the sample to bring its moisture content near to a level which will not bias the results of further testing or sample preparation

3.1.34

routine sample preparation

〈bulk material〉 sample preparation carried out by the stipulated procedures in the specific International Standard in order to determine the average quality of the lot

3.1.35

non-routine sample preparation

〈bulk material〉 sample preparation carried out for experimental sampling

3.1.36

nominal top size

〈bulk material〉 particle size expressed by the aperture dimension of the test sieve (from a square hole sieve series complying with ISO 565) on which no more than 5 % of the sample is retained

3.1.37

nominal bottom size

〈bulk material〉 particle size expressed by the aperture dimension of the test sieve (from a square hole sieve series complying with ISO 565) through which no more than 5 % of the sample passes

3.1.38

quality variation

〈bulk material〉 standard deviation of the quality characteristics determined either by estimating the variance between interpenetrating samples taken from the lot or sub-lot, or by estimating the variance from a variographic analysis of the differences between individual increments separated by various lagged intervals

3.1.39**sampling procedure**

〈bulk material〉 operational requirements and/or instructions relating to taking increments and constituting a sample

3.1.40**sample preparation procedure**

〈bulk material〉 operational requirements and/or instructions relating to methods and criteria for sample division

3.1.41**sampling scheme**

〈bulk material〉 specification of the type of sampling to be used combined with the operational specification of the entities or increments to be taken, the samples to be constituted and the measurements to be made

EXAMPLE The scheme may specify, for example, that the sampling shall be systematic and in two stages. In combination with the specification of the type of sampling, the scheme, in this example, also may specify the number of increments to be taken from a lot, the number of composite samples (or gross samples) per lot, the number of test samples per composite sample, and the number of measurements per test sample.

3.1.42**sampling system**

〈bulk material〉 operational mechanism and/or mechanical installation for taking increments and sample preparation

3.2 Symbols

A list of symbols used in this part of ISO 11648 is presented in Table 1 with short descriptions of symbol meanings and references to the subclauses where the symbols are first mentioned. Table 2 gives a list of subscripts with their meanings that are used in this part of ISO 11648.

Table 1 — Symbols

Symbol	Meaning	Units	First mention
A_{cor}	random component of variance of the corrected variogram and equal to its intercept	—	5.3.2
A_{der}	derived variogram intercept for the increment mass to be used for sampling	—	8.2.2
A_F	constant used to calculate the fundamental error component of the variogram intercept and with the units of a density	kg/mm ³ or kg/m ³ × 10 ⁻⁹	5.3.2
A_0	constant derived from a least squares fit to between-sample variance data	—	9.2.2
a_i	measurement of the quality characteristic of the test sample prepared from sub-lot sample A_i	—	5.3.3
B	gradient (i.e. slope) of variogram, for mass-basis sampling, or for time-basis sampling	min ⁻¹ (time) t ⁻¹ (mass)	5.3.2
b	effective aperture width of cutter	mm	13.3.4
b_i	measurement of the quality characteristic of the test sample prepared from sub-lot sample B_i	—	5.3.3

Table 1 — Symbols (*continued*)

Symbol	Meaning	Units	First mention
$b_{\min}$	minimum cutting aperture width	m	7.2
d	nominal top size of particles	mm	5.3.2
d_L	lower size limit and defined as the finest sieve aperture width that passes 5 % of the undersized particles	mm	9.2.3
d_λ	nominal top size at which complete liberation occurs	mm	9.2.3
e_{del}	increment-delimitation error	—	5.2.1
e_E	increment-extraction error	—	5.2.1
e_F	fundamental error	—	5.2.1
e_G	segregation and grouping error	—	5.2.1
e_P	preparation error (also known as accessory error)	—	5.2.1
e_{Q1}	short-range quality fluctuation error	—	5.2.1
e_{Q2}	long-range quality fluctuation error	—	5.2.1
e_{Q3}	periodic quality fluctuation error	—	5.2.1
e_T	total sampling error	—	5.2.1
e_W	weighting error	—	5.2.1
f_{comp}	mineralogical composition factor	t/m ³	9.2.3
f_r	size range factor	—	9.2.3
f_s	particle shape factor	—	9.2.3
H	heterogeneity index of the bulk material	—	9.2.4
H_S	heterogeneity index for the size range S of the bulk material	—	9.2.4
i	index designating the number of an increment or sub-lot depending on context	—	5.3.2
J	total number of particles in the experimental method for determining fundamental error	—	9.2.4
j	index designating the number of a particle in the experimental method for determining fundamental error	—	9.2.4
k	number of increments defining the lag of a variogram value; or in 5.4, the number of sub-lot samples	—	5.3.2
m_g	gross sample mass	kg	5.3.2
m_I	increment mass	kg	5.3.2
m_{lot}	total mass for lot	t	5.3.2
m_H	estimate of the mass of particles in the size range $d/2$ to d and used to calculate the heterogeneity index	kg	9.2.4
m_{sel}	combined dry mass of the particles selected in the method to determine the heterogeneity index	kg	9.2.4
m_{IS}	increment mass to be used for routine sampling	kg	8.2.2
m_{sub}	mass of the sub-lot	t	8.2.1

Table 1 — Symbols (continued)

Symbol	Meaning	Units	First mention
m_1	mass of container plus lid plus material test portion	kg	20.4.2
m_2	mass of drying tray	kg	20.4.2
m_3	mass of dry container plus lid plus drying tray plus material test portion	kg	20.4.2
m_4	mass of dry empty container	kg	20.4.2
n	number of increments	—	5.3.2
n_I	number of increments comprising each subsample A_i or B_i	—	5.3.3
n_{lot}	minimum number of increments for the lot		16.5
n_{sub}	number of increments taken from each sub-lot	—	16.2
q	flow rate of bulk material stream	t/h	7.2
R_i	range of paired measurements	—	5.3.3
$\bar{R}$	average of the ranges R_i	—	5.3.3
r	number of replicate determinations	—	5.4
s_{BS}^2	between-sample variance	—	9.2.2
s_{comp}^2	composition variance of a unit mass increment	—	5.3.2
s_D^2	distribution variance	—	5.3.2
s_F^2	fundamental error variance	—	5.3.2
s_G^2	segregation and grouping error variance	—	5.3.2
s_I^2	primary increment variance	—	5.3.3
s_{Iunc}^2	uncorrected increment variance	—	5.3.3
s_M^2	measurement (or analysis) variance	—	5.4
s_P^2	sample preparation variance	—	5.4
s_{PM}^2	sample preparation and measurement variance	—	5.3.3
s_{Q1}^2	short-range quality fluctuation variance	—	5.3.1
s_{Q2}^2	long-range quality fluctuation variance	—	5.3.1
s_{rel}^2	relative variance	—	9.2.4
s_S^2	sampling variance	—	5.3.1
s_{S1}^2	primary sampling variance	—	5.4
s_{S2}^2	secondary sampling variance	—	5.4
s_{S3}^2	tertiary sampling variance	—	5.4
s_{sub}^2	sub-lot variance	—	16.5

Table 1 — Symbols (*continued*)

Symbol	Meaning	Units	First mention
s_{SPM}^2	overall variance	—	5.4
s_{WSL}^2	within-sub-lot variance	—	5.3.3
t	lag value for calculating the variogram either on a time or mass basis	min (time) t (mass)	5.3.2
t_{lot}	total time for sampling the lot	min	5.3.2
t_{sub}	total time for sampling the sub-lot	min	5.3.2
u_{lot}	number of sub-lots in the lot	—	16.5
u_{sub}	number of sub-lots actually sampled in an intermittent sampling scheme	—	16.5
V_{cor}	value of corrected variogram	—	5.3.2
V_{exp}	value of experimental variogram	—	5.3.2
v_{cut}	maximum cutter speed of sampler	m/s	7.2
v_{B}	speed of conveyor belt	m/s	7.3
w_{k}	percentage by mass of key component	% by mass	9.2.3
w_{m}	air-dried moisture content of the test portion	% by mass	20.4.2
x_i	value of quality characteristic for increment i	—	5.3.2
x_{m}	mass-weighted average of the quality characteristic	—	9.2.4
$\bar{x}$	mean quality characteristic for all increments	—	5.3.3
β_{SPM}	total (or overall) precision	—	5.4
Δm	mass interval between increments	t	5.3.2
Δt	time interval between increments	min	5.3.2
λ	liberation factor	—	9.2.3
ρ	bulk density of material	t/m ³	7.4
ρ_{k}	density of particles of key component	t/m ³	9.2.3
ρ_{nk}	density of particles of non-key component	t/m ³	9.2.3
$\hat{\sigma}_{\text{F}}^2$	estimate of the fundamental error variance	—	9.2.3

Table 2 — Subscripts

Subscript	Meaning
B	conveyor belt
BS	between-sample
comp	composition
cor	corrected value
cut	cutter
del	delimitation
der	derived
D	distribution
E	extraction
e	experimental
F	fundamental
G	segregation and grouping
g	gross
<i>H</i>	heterogeneity index
<i>i</i>	index designating the number of an increment or sub-lot depending on context
I	increment
Iunc	uncorrected increment
<i>j</i>	index designating the number of a particle in the experimental method for determining fundamental error
k	key component
L	lower size limit
lot	lot
M	measurement
m	air-dried moisture content
<i>m</i>	mass-weighted average
min	minimum
nk	non-key
P	sample preparation
PM	sample preparation and measurement
Q1	short-range quality
Q2	long-range quality
Q3	periodic quality
r	size range of sieve aperture widths
rel	relative
S	size range

Table 2 — Subscripts (*continued*)

Subscript	Meaning
S	sampling
S1	primary sampling
S2	secondary sampling
S3	tertiary sampling
SPM	sampling, sample preparation and measurement (= total or overall)
s	particle shape
sel	particles selected
sub	sub-lot
T	total sampling
W	weighting
wsI	within-sub-lot
λ	liberation factor

4 Applications of bulk material sampling

This part of ISO 11648 provides guidelines for sampling, sample preparation and testing of particulate materials in bulk for a wide range of applications. Typical examples of quantities in bulk are a cargo of coal or iron ore from a single source, a partial cargo (a few cargo spaces) of concentrate or fertilizer, a barge with cement, a unit train with grain, and so on. Quantities in bulk that are defined on a time basis are less commonly specified, but examples of such quantities in bulk are masses or volumes produced during certain time intervals (e.g. shifts or 24-h periods).

Generally, a quantity of material in bulk is inspected and evaluated by collecting a set of unbiased primary increments, preparing test samples (for physical properties and/or chemical composition) without introducing a bias, and analysing test portions by applying approved test methods with properly calibrated apparatus. Adjectives are often added to describe test samples. For example, the terms “chemical analysis sample, moisture sample, size sample”, and other types of samples, are used in various ISO standard methods. The term “test portion” is used in reference to the sample mass or volume taken from test samples for testing or analysis.

Under certain conditions, the effect of a serial correlation on the sampling variance can be taken into account to refine precision estimates and to optimize statistical process control. For many materials, primary increments collected at short intervals (typically 10 min or less) are often correlated. If a sequence of measurements is correlated in space or time, variability between the serial measurements is overstated by the overall variance. On-line analysers are useful to test sets of consecutive measurements for serial correlations.

However, for most manual sampling regimes and many mechanical sampling systems, the sampling interval is too long to show a significant serial correlation between consecutive increments. Thus, the time series variance increases as a function of the sampling interval until it becomes equal to the variance of the measurements for the set of increments collected. At that spacing between primary increments, the measurements are no longer correlated in time, but have become statistically independent^[2].

Duplicate test portions provide an estimate for the analytical variance, often as a function of the variable of interest. The variances between randomly distributed and ordered data sets, and the analytical variance, can be used to optimize sampling regimes for a quantity in bulk of a given intrinsic variability.

5 Principles of sampling

5.1 General

Sample inspections or evaluations of bulk particulate materials for a single lot are normally based on collecting a set of unbiased primary increments from the lot (cargo or shipment), preparing a test sample from the lot sample without introducing a systematic error, and taking a test portion from the test sample and analysing it by applying an appropriate and properly calibrated analytical method or test procedure under prescribed conditions. The precision of the variable of interest in valuable bulk materials can be estimated by dividing a shipment into four or more lots, and by selecting a pair of interpenetrating primary samples of each lot.

NOTE Statistical concepts used in this part of ISO 11648 to describe uncertainty in the measurements on samples, such as bias, precision and variance, are described in the *Guide to the expression of uncertainty in measurement*.

The objective of the operational sequence of sample collection, test sample preparation and analysis of test portions is to estimate the variable of interest in an unbiased manner, with an acceptable and affordable degree of precision. The general sampling theory, which is based on the additive property of variances, can be applied to describe how the variances of sampling, preparation and chemical analysis or physical testing cumulate to determine the total variance.

If a sampling scheme is to ensure correct selection probabilities, it is a requirement that all parts of the bulk particulate material in the lot have an equal opportunity of being selected and appearing in the lot sample for testing. Thus a quantity of material in bulk should be sampled in such a way that all possible primary increments in the set into which the quantity can be divided have a uniform finite probability to be selected. Any deviation from this basic requirement can result in an unacceptable bias. A sampling scheme with incorrect selection techniques (i.e. with non-uniform selection probabilities) cannot be relied upon to provide representative samples.

The objective of this part of ISO 11648 is to describe a common and consistent approach for collecting a set of primary increments from a lot of bulk particulate material (clauses 5 to 16) and for preparing one or more test samples without introducing a systematic error (clauses 17 to 23).

In a strict interpretation of the term, a lot of bulk material is always three dimensional. However, in many practical circumstances, two of the dimensions of the lot can be regarded as being of secondary importance. In industrial operations requiring bulk material transportation mechanisms such as conveyor belts and falling streams at transfer points, the lot can be well described by a one-dimensional model. Dynamic sampling using a stream cutter or static sampling from a stopped conveyor belt can be performed to collect representative samples from such a lot. Clauses 5 to 15 relate to dynamic and static sampling on lots regarded as being one dimensional.

In some situations, static sampling from three-dimensional lots such as stockpiles, ships' holds and wagons cannot be avoided, but it presents considerably more difficult sampling problems, and the risk of collecting unrepresentative samples is considerable. Clause 16 provides sampling procedures for sampling from three-dimensional lots.

Sampling of one-dimensional lots should preferably be carried out by stratified systematic sampling (see 5.3.2), either on a mass basis (see clause 10) or on a time basis (see clause 11). However, it needs to be shown that no systematic error (or bias) can possibly be introduced by periodic variation in quality or quantity when the proposed sampling interval is approximately equal to a multiple of the period of variation in quality or quantity.

As an example, a primary cutter may be cutting a stream of ore which is being reclaimed from a stockpile by a bucket-wheel reclaimer. At both limits of the bucket-wheel traverse across the material interface on the stockpile, the material may have different properties from that of the stockpile average (e.g. due to segregation, surface drying, oxidation, addition of dust-suppressing chemicals, or water sprays). In this case, it is quite possible that every time the primary cutter makes a cut, the cut coincides with ore being delivered from the limit of a traverse of the bucket-wheel reclaimer. Thus, a systematic error can occur.

When a systematic error is introduced at the primary sampling stage by periodic variation in quality or quantity of the particulate material, or where it is felt that, owing to the manner in which the material is handled and presented to subsequent division apparatus, a systematic error can occur in the secondary and subsequent stages of division, it is strongly recommended that stratified random sampling within fixed time or mass intervals be carried out (see clause 12).

The methods for sampling and sample preparation depend on the final choice of sampling scheme and on the steps necessary to minimize possible systematic errors arising during subsequent division steps.

5.2 Sampling errors

5.2.1 General

The processes of sampling, sample preparation and measurement are experimental procedures. As a consequence of these procedures, each process has its own element of uncertainty which results as random variations in the values measured. These random variations which average out to be a negligible value are called experimental errors. A more adverse component contributing to the uncertainty of results are systematic errors appearing as random variables which average out to a biased value away from zero. There is also the category of human errors which are variations that result from departures from the prescribed procedures and which are not random variables suited to statistical analysis.

The total sampling error e_T can be expressed as the sum of a number of independent components^[1]. However, such a simple additive combination is not appropriate for components which are correlated. The total sampling error, expressed as a sum of its components, is:

$$e_T = e_{Q1} + e_{Q2} + e_{Q3} + e_W + e_{del} + e_E + e_P \quad (1)$$

where

- e_{Q1} is the short-range quality fluctuation error associated with short-range variations in quality;
- e_{Q2} is the long-range quality fluctuation error associated with long-range variations in quality;
- e_{Q3} is the periodic quality fluctuation error associated with periodic variations in quality;
- e_W is the weighting error associated with variations in flow rate;
- e_{del} is the increment-delimitation error introduced by incorrect increment delimitation;
- e_E is the increment-extraction error introduced by incorrect increment extraction;
- e_P is the preparation error (also known as accessory error) introduced by departures (usually unintentional) from correct practices for handling of the sample.

The short-range quality fluctuation error, e_{Q1} , consists of two components, as shown by the following equation:

$$e_{Q1} = e_F + e_G \quad (2)$$

where

- e_F is the fundamental error due to variation in quality between particles;
- e_G is the segregation and grouping error.

The fundamental error results from the composition heterogeneity of the lot; that is, the heterogeneity that is inherent to the composition of each particle making up the lot. The greater the differences in the compositions of particles, the greater the composition heterogeneity, and the higher the composition variance. The fundamental error can never be completely eliminated. It is an inherent error resulting from the variation in composition of the particles of the material being sampled.

The segregation and grouping error results from the distribution heterogeneity of the sampled material^[3]. The distribution heterogeneity of a lot is the heterogeneity arising from the manner in which particles are scattered in the bulk material.

A number of the components of the total sampling error, namely e_{del} , e_{E} and e_{P} , can be made negligible by using correct sampling practices. The remainder can be minimized, or reduced to an acceptable level, by correct design of the sampling procedure.

5.2.2 Preparation error, e_{P}

In this context, the preparation error includes error associated with non-selective sample preparation operations that should not change mass, such as sample transfer, drying, crushing, grinding or mixing. It does not include errors associated with sample division or testing. Preparation errors, also known as accessory errors, include sample contamination, loss of sample material, alteration of the chemical or physical composition of the sample, operator mistakes, fraud or sabotage. These errors can be made negligible by correctly designing the plant sampling and training staff. For example, the cutters should have dust caps to prevent entry of dust when the cutter is in the parked position and moist samples should be prepared as quickly as possible to avoid loss of moisture content due to evaporation. If samples are to be extracted for use in size analysis, excessive vertical drops should be avoided, because breakage of coarse particles will alter the physical characteristics of the sample.

5.2.3 Delimitation and extraction errors, e_{del} and e_{E}

Delimitation and extraction errors arise from incorrect sample-cutter design and operation. The increment-delimitation error, e_{del} , results from an incorrect shape of the volume delimiting the increment, and this can be due to both faulty design and operation. Sampling with non-uniform selection probabilities results from an incorrect shape of the increment volume. The mean of e_{del} is often different from zero, which makes it a source of sampling bias. The delimitation error can be made negligible if all parts of the stream cross-section are diverted by the sample cutter for the same length of time.

The increment-extraction error, e_{E} , results from incorrect extraction of the increment. The extraction is said to be correct if, and only if, all particles with the centre of gravity inside the boundaries of the correctly delimited increment are extracted. The mean of e_{E} is often different from zero, which also makes it a source of sampling bias. The extraction error can be made negligible by ensuring that the increment is completely extracted from the stream without any material rebounding or being lost from the cutter.

5.2.4 Weighting error, e_{W}

The weighting error is an error component arising from the selection model underlying equation (1). In the model, the time-dependent flow rate of the particulate material stream is a weighting function applied to the time-dependent quality characteristic in the integral over time which evaluates the mean quality characteristic of the lot. The weighting error represents the error due to the amplifying effect of the flow-weighting function on the quality characteristic fluctuations. The best solution to reducing the weighting error is to stabilize the flow rate. As a general rule, the weighting error is negligible for flow-rate variations not exceeding 10 %, and acceptable for flow-rate variations not exceeding 20 %.

5.2.5 Periodic quality fluctuation error, e_{Q3}

Periodic quality fluctuation errors result from periodic variations in quality generated by certain equipment used for bulk-material handling, for example crushing and screening circuits and bucket-wheel reclaimers. The presence of periodic variations can be detected by determining the variogram (see 5.3.1). While in most cases variogram values can be fitted with a simple linear or quadratic function, if periodic behaviour (characterized by regularly spaced maxima and minima) is observed, the fitting function can include a sine wave term with a period and an amplitude to be determined as parameters of the fit^[1]. In such cases, stratified random sampling should be carried out as discussed in 5.1. The alternative is to significantly moderate the source of periodic variations in quality, which may require the redesign of plant systems.

5.3 Sampling variance

5.3.1 General

Assume that the weighting, increment-delimitation, increment-extraction and accessory errors ($e_W + e_{del} + e_E + e_P$) have been eliminated or reduced to insignificant values by careful design and sampling practice. Furthermore, assume that periodic variations in quality have been eliminated and that the flow rate has been regulated. The sampling error in equation (1) is reduced to:

$$e_T = e_{Q1} + e_{Q2} \quad (3)$$

Hence, the sampling variance s_S^2 is given by:

$$s_S^2 = s_{Q1}^2 + s_{Q2}^2 \quad (4)$$

The short-range quality fluctuation variance, s_{Q1}^2 , arises from the different internal composition of increments taken at the shortest possible interval apart. This is a local or random variance due to the particulate nature of the bulk material.

The long-range quality fluctuation variance, s_{Q2}^2 , arises from the continuous trends in quality that occur while sampling a bulk material and is usually space- and time-dependant. This component is often the combination of a number of trends generated by diverse causes.

5.3.2 Estimation of sampling variance from the variogram

In this method, the short-range and long-range quality fluctuation variances are determined from a time series analysis of a statistical experiment in which a large number (e.g. 20 to 40) of individual increments are taken in sequence from a lot, processed in accordance with the methods given in this part of ISO 11648 and the quality characteristics of each increment analysed in duplicate.

The differences between the quality characteristic values measured for successive pairs separated by k increments in the sequence are then calculated. As shown in equation (5), the squared differences are summed and divided by the number of increment pairs that can be formed having the specified separation (lag). The variance value $V_{exp}(t)$ so formed by this pooling is the serial variance for the lag of k increments. The plot of the serial variance versus lag is called a variogram. It is closely related to the auto-covariance function used in signal analysis and other engineering applications of time series analysis. A statistical experiment of this kind, for which the objective is to construct a variogram, is often called a variographic experiment. An example of a variographic experiment and the construction and plotting of a variogram are shown in annex A.

In mathematical notation, the serial variance $V_{exp}(t)$ corresponding to a lag of k increments is given by the following equation:

$$V_{exp}(t) = \frac{\sum_{i=1}^{n-k} (x_{i+k} - x_i)^2}{2(n-k)} \quad (5)$$

where

x_i is the value of the quality characteristic for increment i ($i = 1, 2, \dots, n$);

$n - k$ is the number of pairs of increments at integer lag k apart;

t is equal to $k\Delta t$, where Δt is the sampling interval in units of time (expressed in minutes) or to $k\Delta m$ where Δm is the sampling interval in units of mass (expressed in tonnes), depending on whether time-basis or mass-basis sampling is used.

The term $(n - k)$ in the denominator of equation (5) reflects the degrees of freedom for the variance term at the specified interval k , while the factor of 2 in the denominator ensures that, as $t \rightarrow 0$, $V_{\text{exp}}(t)$ tends to the conventional variance of measurements, taken at the same position.

The resultant variogram, $V_{\text{exp}}(t)$, is called the “experimental” variogram, and includes the variance of sample preparation and measurement as well as the sampling variance. If the extracted increments are prepared and analysed in duplicate, the sample preparation and analysis variance can be determined in accordance with the procedures described in ISO 3084. Subtraction of the sum of the sample preparation and analysis variances ($s_P^2 + s_M^2$) from the calculated value of $V_{\text{exp}}(t)$ at each lag gives the “corrected” variogram, $V_{\text{cor}}(t)$, which provides information on the sampling variance only. However, caution should be observed when subtracting the sample preparation and analysis variance from the serial variance values of the experimental variogram. The difference between the serial variance and the sample preparation and analysis variance is only a valid estimate for the sampling variance if the F -ratio between these variances is statistically significant.

Variograms that occur in practice can usually be approximated in the range $0 \leq t \leq 4\Delta t$ by a straight line. The two coefficients of the straight line (intercept A_{exp} and slope B) shall be determined by a linear least squares fit to the experimental variogram values of the first four lags.

NOTE The variographic method for determining the sampling variance is applicable to the sampling of a stream of bulk material from one production source, but when a lot may consist of sub-lots from different sources, difficulties are encountered.

Thus, it can be assumed that, in the range $0 \leq t \leq 4\Delta t$, an acceptable approximation to the corrected variogram is the linear function:

$$V_{\text{cor}}(t) = A_{\text{cor}} + B \cdot t = (A_{\text{exp}} - s_P^2 - s_M^2) + B \cdot t \quad (6)$$

where

A_{cor} is the random component of variance of the corrected variogram;

A_{exp} is the experimental intercept of the experimental variogram;

B is the gradient (or slope) of the variogram. B is expressed in units of inverse mass (expressed in tonnes⁻¹) for mass-basis sampling, or inverse time (expressed in min⁻¹) for time-basis sampling;

s_P^2 is the sample preparation variance;

s_M^2 is the measurement (or analysis) variance.

It should be noted that the linear approximation to the variogram based on a four-point fit is subjective to the extent that taking more or less points will result in a different linear fit. This includes the procedure where a line is passed through the first two points. The estimates of the parameters A_{cor} and B and the variances derived from these parameters are also subjective to the same extent.

The sampling variances, s_S^2 , for stratified systematic sampling and stratified random sampling have been shown to be related to the coefficients A_{cor} and B of the linear approximation. Equations (7) and (8) given below for sampling variances are derived by establishing a mathematical relationship between the variogram values and the variance of the estimation error between the sample mean and the population mean^[1].

a) Stratified systematic sampling:

$$s_S^2 = \frac{A_{\text{cor}}}{n} + \frac{B \cdot m_{\text{lot}}}{6n^2} \quad (7)$$

where

n is the number of increments;

m_{lot} is the total mass, expressed in tonnes, of the lot.

Strictly, this equation is only precise for centralized systematic sampling, where the increment is taken from the centre of each stratum. However, in practice, it is a close approximation to the sampling variance for non-central systematic sampling.

b) Stratified random sampling:

$$s_S^2 = \frac{A_{\text{cor}}}{n} + \frac{B \cdot m_{\text{lot}}}{3n^2} \quad (8)$$

NOTE Equations (7) and (8) apply to mass-basis sampling. For time-basis sampling, m_{lot} is replaced by t_{lot} , the total time, expressed in minutes, for sampling the lot.

Thus, where there are no periodic variations in quality, systematic sampling is more precise than stratified random sampling.

In Equations (7) and (8), the first and second terms correspond to the short-range and long-range quality fluctuation variances respectively. Thus, for stratified systematic sampling:

$$s_{Q1}^2 = \frac{A_{\text{cor}}}{n} ; \text{ and} \quad (9)$$

$$s_{Q2}^2 = \frac{B \cdot m_{\text{lot}}}{6n^2} \quad (10)$$

The variogram intercept A_{cor} is made up of two components^[1]:

- the segregation and grouping error variance s_G^2 ;
- the fundamental error variance s_F^2 for the particular increment mass used for the variographic experiment.

The fundamental error variance for the increment mass results from the particle-to-particle variation in quality of the material making up the increment; it can be determined as described in clause 9. The fundamental error variance for the increment mass is proportional to the cube of the nominal top size and inversely proportional to the increment mass. Thus, the variogram intercept A_{cor} can be expressed as follows:

$$A_{\text{cor}} = s_G^2 + \frac{A_F \cdot d^3}{m_I} \quad (11)$$

where

A_F is a constant with the same units as density (expressed in kilograms per cubic millimetre, or kilograms per cubic metre $\times 10^{-9}$);

d is the nominal top size, expressed in millimetres, of the particles;

m_I is the increment mass, expressed in kilograms.

If the results of a variographic experiment are to be applied to sampling with a different increment mass, it is necessary to determine A_F (see clause 9).

For stratified systematic sampling, combining the above equations gives:

$$s_S^2 = \frac{A_F \cdot d^3}{m_g} + \frac{s_G^2}{n} + \frac{B \cdot m_{lot}}{6n^2} \quad (12)$$

where

$\frac{A_F \cdot d^3}{m_g}$ is the fundamental error variance for the gross sample mass;

m_g is the gross sample mass, expressed in kilograms, and is equal to $n \cdot m_I$.

Hence, the fundamental error component of the sampling variance for the gross sample mass is determined by the nominal top size of the bulk material and the gross sample mass.

NOTE While the methods of this part of ISO 11648 are likely to be satisfactory for many bulk materials other than ores, mineral concentrates, coal, and industrial chemicals in particulate form, caution should be observed when applying relationships such as that between the fundamental error variance, particle size and gross sample mass to materials with particle shapes and densities markedly different from minerals, for example, to wood chips or bulk tea. In these cases, fully experimental methods should be used and supported by a program of test work (see 9.2.2).

EXAMPLE An example showing the use of the variogram intercept A_{cor} and the variogram slope B to calculate the sampling variance is given in A.4 of annex A.

The quantities:

$$A_F \cdot d^3 \quad \text{and} \quad s_G^2 + \frac{B \cdot m_{lot}}{6n}$$

are referred to by some authors as the “composition variance of a unit mass increment” and the “distribution variance” respectively.

Then:

$$s_S^2 = \frac{s_{comp}^2}{m_g} + \frac{s_D^2}{n} \quad (13)$$

where

s_{comp}^2 is the composition variance of a unit mass increment;

s_D^2 is the distribution variance.

Note that for a “flat” variogram, where $B = 0$, the distribution variance, s_D^2 , is equivalent to the grouping error variance, s_G^2 .

5.3.3 Alternative methods of estimating sampling variance

The variographic method suffers to some extent from the subjectivity inherent in approximating the variogram at small lag values with a linear function. Ambiguous variograms are sometimes encountered which give wide variations for slope and intercept depending on the number of variogram values included in the least squares fit. However, the variographic method in which sampling variance is estimated from the experimental variogram is more rigorous than others available.

There are two simpler methods, each having the advantages of being easier and less costly to apply than the variographic method. These are the increment variance method and the within-strata variance method. The increment variance method provides a better margin of safety.

However, unlike the variographic method, each of these alternative methods has the disadvantage of not being able to determine the separate contributions of the short-range and long-range fluctuation error variances, i.e.:

$$s_{Q1}^2 \text{ and } s_{Q2}^2$$

Consequently, if the sampling variance needs to be reduced, these methods do not show whether the mass of individual increments or the number of increments needs to be increased. In contrast, the variogram method immediately provides this information.

The alternative methods are as follows.

a) Increment variance

The uncorrected increment variance, s_{Iunc}^2 , biased by the preparation and measurement variance is defined as the variance of the quality characteristics of all increments taken from the lot, i.e.:

$$s_{\text{Iunc}}^2 = \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2 \quad (14)$$

where

n is the number of increments;

x_i is the value of the quality characteristic for increment i ;

$\bar{x}$ is the average of the quality characteristic measured on all increments.

The sample preparation and measurement variance, s_{PM}^2 , which should be determined by duplicate preparation and testing on each increment as in the variographic method, is subtracted from the uncorrected increment variance to obtain the primary increment variance, s_{I}^2 .

The estimated sampling variance is then given by:

$$s_{\text{S}}^2 = \frac{s_{\text{Iunc}}^2 - s_{\text{PM}}^2}{n} = \frac{s_{\text{I}}^2}{n} \quad (15)$$

This method overestimates the sampling variance, because it neglects trends and assumes that there is no correlation between adjacent increments.

b) Within sub-lot variance

The within sub-lot variance method involves determining the variance of the desired quality characteristic within sub-lot (s_{WSL}^2) by duplicate sampling in accordance with ISO 3084. In ISO 3084, this variance is called the variance within-strata as the lot is considered to be divided into n approximately equal sub-lots in a partitioning based on mass, time or space (i.e. in strata, where each stratum defines a sub-lot).

The lot is divided into n sub-lots. The procedure for constituting duplicate sub-lot samples for each sub-lot is in accordance with ISO 3084. Test samples are prepared from each sub-lot sample and the quality characteristic measured.

The range, R_i , of paired measurements is given by the equation:

$$R_i = |a_i - b_i| \quad (16)$$

where

a_i is the measurement of quality characteristic of the test sample prepared from sub-lot sample A_i ;

b_i is the measurement of quality characteristic of the test sample prepared from sub-lot sample B_i which pairs with sub-lot sample A_i ;

i is the subscript which designates each sub-lot.

The average of the ranges R_i is given by the equation:

$$\bar{R} = \frac{\sum R_i}{n} \quad (17)$$

where n is the number of R_i , i.e. the number of pairs of sub-lot samples.

For example, 100 increments may be required to be extracted from the lot, and 10 pairs (i.e. $n = 10$) of sub-lot samples, each comprised of five increments, are constituted in accordance with the methods of ISO 3084. Test samples are prepared from each of these 20 sub-lot samples and the quality characteristic measured for each test sample.

The estimated within sub-lot variance, s_{WSL}^2 , in one investigation is given by the equation:

$$s_{WSL}^2 = \frac{\pi}{4} n_1 \bar{R}^2 \quad (18)$$

where

n_1 is the number of increments comprising each subsample A_i or B_i ;

$\pi/4$ is the factor to estimate variance from the range for paired data.

The estimated sampling variance in this case is given by:

$$s_S^2 = \frac{s_{WSL}^2 - s_{PM}^2}{n} \quad (19)$$

However, the value of s_{WSL}^2 obtained depends on the size of the strata used for collecting the data, i.e. the mass or time interval between increments. Consequently, the within sub-lot variance method should be used only where the proposed sampling interval is not greatly different from that used to determine s_{WSL}^2 .

5.4 Total variance and precision

The total (or overall) variance is denoted by s_{SPM}^2 . It comprises three components, namely the variance of sampling, the variance of sample preparation and the variance of measurement, as given in equation (20).

$$s_{\text{SPM}}^2 = s_{\text{S}}^2 + s_{\text{P}}^2 + s_{\text{M}}^2 \quad (20)$$

where

s_{S}^2 is the sampling variance;

s_{P}^2 is the sample preparation variance;

s_{M}^2 is the measurement variance.

The methods for determining estimates of s_{S}^2 may be found in 5.3.2 and 5.3.3 of this part of ISO 11648.

NOTE The distinction between “sampling” and “sample preparation” is not always clear. For the purposes of this part of ISO 11648, “sampling” stages denote those stages of sampling and sample division that take place within the sampling plant where primary increments are extracted and where possibly size reduction, secondary and tertiary division of primary increments are carried out. Whereas, “sample preparation” stages denote those stages that take place away from the sampling plant, typically in the plant laboratory. The principles of sampling given in 5.3 apply to sample preparation stages as well as to the sampling stages.

The total (or overall) precision, β_{SPM} , is a measure of the combined precision of sampling, sample preparation, and measurement. For a symmetrical two-sided confidence interval of 95 % and where the number of independent comparisons (degrees of freedom) that can be made among the set of measurements is large.

$$\beta_{\text{SPM}} = 1,96 s_{\text{SPM}}$$

In practice, the approximation:

$$\beta_{\text{SPM}} = 2s_{\text{SPM}} = 2\sqrt{s_{\text{S}}^2 + s_{\text{P}}^2 + s_{\text{M}}^2} \quad (21)$$

is often used in bulk materials sampling standards.

Where secondary and tertiary division of primary increments is carried out, the sampling variance can be split into a number of parts as follows:

$$s_{\text{S}}^2 = s_{\text{S}1}^2 + s_{\text{S}2}^2 + s_{\text{S}3}^2 \quad (22)$$

where

$s_{\text{S}1}^2$ is the primary sampling variance;

$s_{\text{S}2}^2$ is the secondary sampling variance;

$s_{\text{S}3}^2$ is the tertiary sampling variance.

Again, the principles of 5.2 apply to each stage. Separate experiments are required to establish the magnitude of each component. Such experiments are useful for identifying the major sources of variance. Splitting the sample variance into its components can also assist in the design of sampling equipment. On the other hand, if all increments are processed in the same manner and only the total sampling variance is required, there is no need to separate the components.

Where a very precise result is required and the sampling variance has been minimized, consideration has to be given to increasing the number of sample preparations and measurements, to reduce these components of the overall variance.

This is achieved by:

- carrying out multiple determinations on the gross sample;
- analysing individual increments (see Figure 1); or
- dividing the lot into a number of sub-lots and analysing a sample from each sub-lot (see Figure 2).

The overall variance in each case is then given by one of the following equations:

- a) where a single gross sample is constituted from a lot and r replicate determinations are carried out on the gross sample:

$$s_{\text{SPM}}^2 = s_{\text{S}}^2 + s_{\text{P}}^2 + \frac{s_{\text{M}}^2}{r} \quad (23)$$

- b) where k sub-lot samples are prepared, each constituted from an equal number of increments, and r replicate determinations are carried out on each sub-lot sample:

$$s_{\text{SPM}}^2 = s_{\text{S}}^2 + \frac{s_{\text{P}}^2 + \frac{s_{\text{M}}^2}{r}}{k} \quad (24)$$

- c) where all n increments are prepared and a single determination is carried out on each increment:

$$s_{\text{SPM}}^2 = s_{\text{S}}^2 + \frac{s_{\text{P}}^2}{n} + \frac{s_{\text{M}}^2}{n} \quad (25)$$

In each case, the sampling variance is determined from the equations given in 5.3.

NOTE The determination of moisture requires special consideration due to the fact that it is extremely difficult, if not impossible, to retain the integrity of the sample over extended periods of sample collection. In such cases, a bias may occur which can be overcome only by collecting moisture samples at more frequent intervals than may be dictated by a simple calculation of the number of primary increments and sub-lot samples for a given precision. It is therefore recommended that moisture tests be carried out on a number of sub-lot samples, and that the average of the test results be calculated, the average weighted according to the masses of the sub-lots in the case of time-basis sampling, or according to the number of increments in each sub-lot sample in the case of mass-basis sampling. This will reduce any bias in the test result caused by moisture loss (or gain) due to climatic conditions. It will also result in better precision. In exceptional circumstances, where the moisture loss is very rapid, secondary and tertiary division is not permissible, unless the sampling system is totally enclosed and handling is minimized.

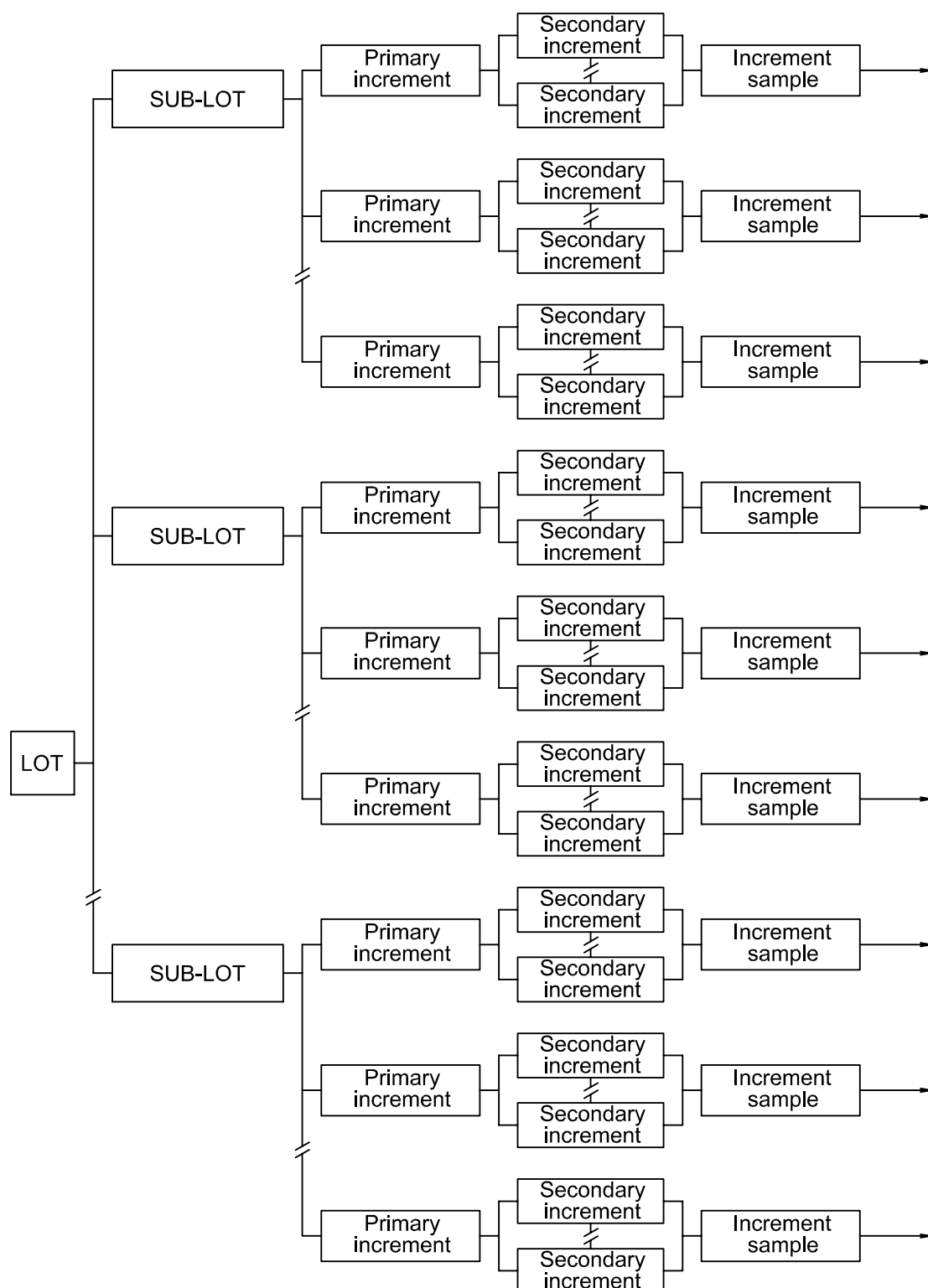
6 Establishing a sampling scheme

Most sampling operations are routine and conform to the definition of regular sampling defined in ISO 11648-1. Regular sampling is sampling carried out by the stipulated procedures in the relevant International Standard in order to determine the average quality of the lot. In establishing a sampling scheme for regular sampling so that a specified precision on a quality characteristic for a lot can be obtained, it is necessary to carry out the following sequence of steps. The sequence includes experimental sampling procedures, such as step g) below, which are not routine and are carried out only infrequently, as, for example, when there is a significant alteration in conditions such as a change in the source of the particulate material or in the sampling equipment.

- a) Define the purpose for which the samples are to be taken. Sampling for the quality verification requirements of commercial transactions is the central purpose within the scope of this part of ISO 11648 and other sampling standards. However, the procedures described in this part of ISO 11648 are applicable to sampling for the purpose of monitoring plant performance and for process control as well.
- b) Identify the quality characteristics to be measured. Specify the total precision (combined precision of sampling, sample preparation and measurement) required for each quality characteristic. It may be found that the required precision gives impractical numbers of primary increments and sub-lots. In such cases, it may be necessary to accept poorer precision.
- c) Define the lot, including its mass or duration.
- d) Define the sub-lots, including their number and their masses or durations.
- e) Ascertain the nominal top size and particle density of the bulk material for use in determining the gross sample mass in step i). The nominal top size also determines the minimum cutter aperture width required to avoid bias where a mechanical sampler is used, or the minimum size of the ladle required to avoid bias where manual sampling is used.
- f) Check that the procedures and equipment for taking increments avoid significant bias (see clause 7).
- g) Determine the variability of the quality characteristics under consideration, using the variogram method or one of the alternatives (see clause 5).
- h) Determine the number of primary increments to be taken from the lot or the sub-lots to be tested (see clause 8).
- i) Determine the minimum gross sample mass (see clause 9).
- j) Determine the sampling intervals, in tonnes for mass-basis systematic sampling (see clause 10) and stratified random sampling within fixed mass intervals (see clause 12), or in minutes for time-basis systematic sampling (see clause 11) and stratified random sampling within fixed time intervals (see clause 12).
- k) Take primary increments at the intervals determined in step j) during the whole period of handling the lot.

In experimental sampling, each increment may be analysed separately (see Figure 1) to assess the variability of the quality characteristic in the lot by monitoring the variogram. Or the primary increments may be taken from a sub-lot (see 10.5 or 11.5) to constitute a sub-lot sample which may also be analysed to assess lot variability (see Figure 2). These are only two of a variety of other experimental sampling schemes possible (see, for example, the fully-nested and staggered-nested experiments described in part 1 of this International Standard, i.e. ISO 11648-1).

In regular sampling, a typical sampling scheme is to combine sub-lot samples so as to constitute a gross sample for analysis (an example is given in Figure 3). Periodically, checks should be made on the precision achieved by the sampling scheme by means of replicate sampling, i.e. by replication of the gross sample. For example, if duplicate sampling is used, each alternate primary increment is diverted so that gross samples A and B are formed (see Figure 4) from which two test samples are prepared and tested.



NOTE Each increment sample is prepared and analysed separately.

Figure 1 — Example of a scheme for experimental sampling with each increment analysed separately

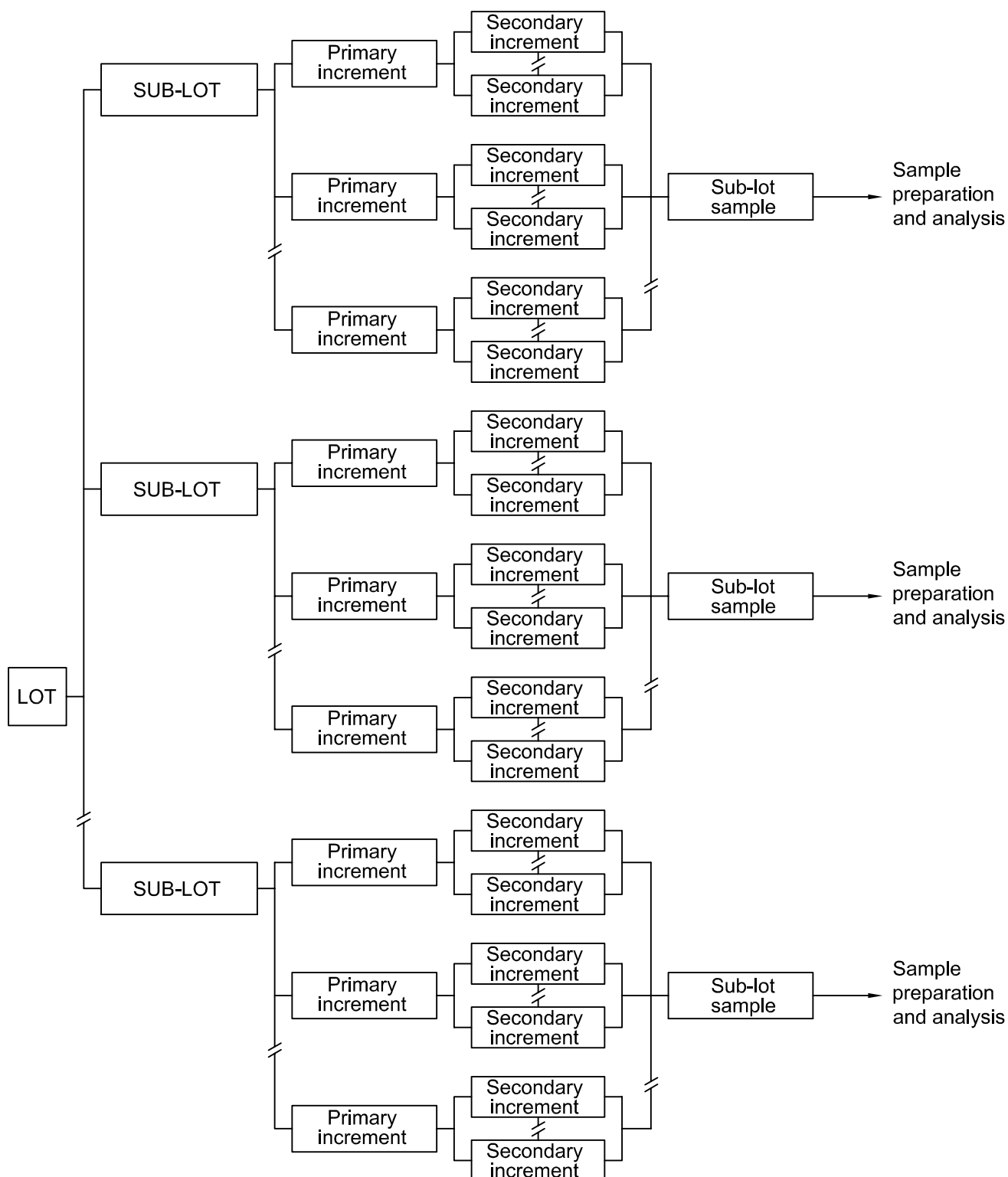


Figure 2 — Example of a scheme for experimental sampling with each sub-lot sample analysed separately

