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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 particulaires

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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