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Statistical aspects of sampling from bulk materials —

Part 1:
General principles

Aspects statistiques de l'échantillonnage des matériaux en vrac —

Partie 1: Principes généraux

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 11648-1 was prepared by Technical Committee ISO/TC 69, *Applications of statistical methods*.

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 under this general title for the sampling of liquids and gases, if the need exists.

Introduction

This first part of ISO 11648 gives a broad outline of the statistical aspects of sampling from bulk material.

International Standards dealing with the methods for sampling for bulk materials, such as solid fuels, iron ores, etc., have already been published and some of these are being revised by the responsible technical committees. This International Standard provides a source of information for technical terms and sampling techniques for types of bulk materials for which International Standards on sampling have not yet been written. This International Standard may also act as a bridge for mutual understanding of terms and methods between Technical Committees.

Statistical aspects of sampling from bulk materials —

Part 1: General principles

1 Scope

This part of ISO 11648 establishes the general principles for the application and statistical treatment of the sampling of bulk materials. It also provides general guidance and examples for estimating necessary variances and checking precision and bias when the average value of a quality characteristic is investigated. Furthermore, this part of ISO 11648 gives information relating to the statistical analyses of serial data, by the use of variograms and correlograms.

This part of ISO 11648 also defines the basic terms with definitions for the sampling of bulk materials. These terms are necessary for providing a better understanding of sampling techniques as well as making it easier to fulfil requirements.

NOTE Part 2 of ISO 11648 is applicable to particulate materials in bulk.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3534 and the following apply.

NOTE 1 The text ⟨bulk material⟩ shown after terms means the definition given is confined to the field of bulk sampling.

NOTE 2 For further information on definitions, see Annex A.

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 characteristics 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 plan

NOTE 1 In bulk sampling, systematic sampling can be achieved by taking items at fixed distances or after time intervals of fixed length. Intervals can, for example, be based on mass or time. In the case of mass, sampling units or increments should be of equal mass. With respect to time, 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 sampling units are characterized by having an equal probability of being selected. Once chosen, the entire sampling unit becomes a part of the whole sample.

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

3.1.10
precision
closeness of agreement between independent test results obtained under stipulated conditions

NOTE 1 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 test results. 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 particulate 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. The error of indication is the

> "indication of a measuring instrument less the 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 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 can 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 plans 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 in mechanical sampling 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 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 plan
⟨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 plan can specify, for example, that the sampling is to be systematic and in two stages. In combination with the specification of the type of sampling, the plan, in this example, also can 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 scheme
⟨bulk material⟩ combination of sampling plans with purposes for sampling

NOTE Purposes for sampling include routine sampling, estimating precision, and investigation of quality variation.

3.1.43
sampling system
⟨bulk material⟩ operational mechanism and/or mechanical installation for taking increments and sample preparation

3.2 Symbols and abbreviated terms

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_i	composite sample of odd increments for the i-th part in interpenetrating sampling	—	7.3
A_2	parameter of significant difference between two means	—	10
B_i	composite sample of even increments for the i-th part in interpenetrating sampling	—	7.3
b	parameter for calculation of limits of confidence interval of variance component	—	B.5
b_0	intercept by linear regression	—	C.5
b_1	gradient (i.e. slope) of linear regression	—	C.5
d	nominal top size of particles	mm	5
d_i	difference between system average and reference average in the same set	—	10
d_2	factor to estimate standard deviation from the range of normally distributed paired data	—	7.3
$\bar{d}$	average difference between system measurements and reference measurements	—	10
$E(V)$	expected variance	—	B.5
F_o	observed F	—	10
$F_{\alpha/2}(v_1,v_2)$	$\alpha/2$-quantile of the F-distribution with v_1, v_2 degrees of freedom	—	10
g_i	difference between x_{i1} and x_{i2}	—	10
h_i	difference between y_{i1} and y_{i2}	—	10
i	index designating the number of an increment or sub-lot depending on context	—	7.3
k	number of increments defining the lag of a variogram or correlogram value, or number of sets of increments	— —	7.4 8
N_{ite}	number of items in a population	—	5
N_{sub}	total number of possible increments in a sub-lot	—	5
n	number of increments	—	6
n_{ite}	number of items in a sample	—	5
n_M	number of measurements of a test sample	—	6
n_o	number of observations in treatment A_i	—	B.5
n_{sub}	number of increments taken from each sub-lot	—	5
P_{mi}	production rate of molten iron	t/tap	C.3
p	number of lots	—	7.2
R_i	range of paired measurements	—	7.3
$\bar{R}$	average of the ranges R_i	—	7.3
$\bar{R}_1$	average of the ranges between duplicate measurements	—	B.4.3
$\bar{R}_2$	average of the ranges between means of measurements in the test samples	—	B.4.3
$\bar{R}_3$	average of the ranges between means of measurements in the composite samples	—	B.4.3
r_{exp}	value of experimental correlogram	—	7.4
S	sum of squares	—	C.7.2
s^2_{BIT}	variance between items	—	5
s^2_d	variance of d_i	—	10

Table 1 *(continued)*

Symbol	Meaning	Units	First mention
s_e^2	error variance	—	10
T_1, T_2	confidence limits	—	10
t	lag value for calculating the variogram or correlogram either on a time or mass basis	min (time), t (mass)	7.4
$t_{(1-\alpha)/2}(v)$	$(1-\alpha)/2$-quantile of t-variable with v degrees of freedom	—	10
U_{CL}	upper control limit	—	D.4
u	unloaded ratio	—	C.5
u_{lot}	number of sub-lots in the lot	—	6
V_{A}	variance with v_{A} degrees of freedom	—	B.5
V_{a}	variance corresponding to the amplitude of cyclic variation	—	C.3
V_{c}	variance of cyclic variation	—	C.3
V_{E}	variance with v_{E} degrees of freedom	—	B.5
V_{exp}	value of experimental variogram	—	7.4
V_{r}	variance of random variation	—	C.3
w_{Al}	percentage by mass of aluminium content	% by mass	C.7
w_{Fe}	percentage by mass of total iron content	% by mass	C.7
w_{m}	percentage by mass of moisture content	% by mass	C.5
w_{sf}	percentage by mass of size fraction	% by mass	C.6
w_{Si}	percentage by mass of silicon content	% by mass	C.3
w_{Su}	percentage by mass of sulfur content	% by mass	C.3
x_i	value of quality characteristic for increment i	—	7.4
x_{i1}	one of the duplicate measurements obtained by a system method	—	10
x_{i2}	one of the duplicate measurements obtained by a system method	—	10
$\bar{\bar{x}}$	grand average of x_{i1} and x_{i2}	—	10
y_{i1}	one of the duplicate measurements obtained by a reference method	—	10
y_{i2}	one of the duplicate measurements obtained by a reference method	—	10
$\bar{\bar{y}}$	grand average of y_{i1} and y_{i2}	—	10
α	level of significance of a test	—	10
δ	maximum tolerable bias	—	10
μ	population mean	—	C.7.2
v	number of degrees of freedom	—	10
ρ_{COD}	parameter of water quality (chemical oxygen demand)	mg/l of oxygen	E.7
σ_{A}^2	variance component between treatments A_i	—	B.5
σ_{BC}^2	variance component between composite samples	—	C.7
σ_{BL}^2	variance component between lots	—	7.2
σ_{BP}^2	variance component between parts	—	C.7
σ_{BT}^2	variance component between trains	—	Annex A

