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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
$\overline{d}$	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
$\overline{R}$	average of the ranges R_i	—	7.3
$\overline{R}_1$	average of the ranges between duplicate measurements	—	B.4.3
$\overline{R}_2$	average of the ranges between means of measurements in the test samples	—	B.4.3
$\overline{R}_3$	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−α)/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
$\overline{\overline{x}}$	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
$\overline{\overline{y}}$	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

Table 1 *(continued)*

Symbol	Meaning	Units	First mention
σ_{BV}^2	variance component between vessels	—	C.7
σ_{BW}^2	variance component between wagons	—	Annex A
σ_{E}^2	expected variance of estimate	—	5
σ_{M}^2	variance component between the measurements obtained on a test sample	—	6
σ_{P}^2	variance component between the test samples prepared from a gross sample	—	6
σ_{S}^2	variance component of sampling	—	7.2
σ_{t}^2	total variance	—	C.3
σ_{wl}^2	variance component within lot	—	8
σ_{wsl}^2	variance component within sub-lot	—	8
σ_{wst}^2	variance component between the increments within stratum in the cases of stratified sampling and systematic sampling, and the variance component between the increments within the valid primary sampling unit in the case of two-stage sampling	—	6
$\hat{\sigma}_{\mathrm{A}}^2$	estimate of variance component of σ_{A}^2	—	B.5
$\hat{\sigma}_{\mathrm{BC}}^2$	estimate of variance component of σ_{BC}^2	—	C.7
$\hat{\sigma}_{\mathrm{BL}}^2$	estimate of variance component of σ_{BL}^2	—	B.5
$\hat{\sigma}_{\mathrm{BP}}^2$	estimate of variance component of σ_{BP}^2	—	C.7
$\hat{\sigma}_{\mathrm{BV}}^2$	estimate of variance component of σ_{BV}^2	—	C.7
$\hat{\sigma}_{\mathrm{E}}$	estimate of standard deviation of σ_{E}	—	D.5
$\hat{\sigma}_{\mathrm{E}}^2$	estimate of variance of σ_{E}^2	—	D.5
$\hat{\sigma}_{\mathrm{M}}^2$	estimate of variance component of σ_{M}^2	—	B.4.3
$\hat{\sigma}_{\mathrm{P}}^2$	estimate of variance component of σ_{P}^2	—	B.4.3
$\hat{\sigma}_{\mathrm{S}}^2$	estimate of variance component of σ_{S}^2	—	B.4.3
$\hat{\sigma}_{\mathrm{wst}}$	estimated standard deviation within-stratum σ_{wst}	—	7.3
$\hat{\sigma}_{\mathrm{wst}}^2$	estimate of variance component of σ_{wst}^2	—	7.3
$\chi^2\left(v,\frac{\alpha}{2}\right)$	$\left(\frac{\alpha}{2}\right)$-quantile of the χ^2-distribution with v degrees of freedom	—	B.5

Table 2 — Subscripts

Subscript	Meaning
Al	aluminium
a	amplitude
BC	between composite samples
BIT	between items
BL	between lots
BP	between parts
BT	between trains
BV	between vessels
BW	between wagons
COD	chemical oxygen demand
c	cyclic
d	difference
E	expectation
e	error
exp	experimental
Fe	iron
ite	item
i	index designating the number of an increment or sub-lot depending on context
L	lower
lot	lot
M	measurement
m	moisture
mi	molten iron
o	observed
P	preparation
r	random
S	sampling
sf	size fraction
Si	silicon
Su	sulfur
sub	sub-lot
t	total
U	upper
wl	within lot
ws	within sample
wsl	within sub-lot
wst	within stratum

4 Purpose and application of statistics in sampling from bulk material

To estimate the amount, or a property or properties of the bulk material, samples are taken from many types of bulk material for various purposes. They may be taken from a continuous stream of material, an individual lot or a sequence of lots. A standard is necessary because of the occurrence of numerous sources of variation within the bulk, due to sampling procedures, as a result of measurement errors and due to the preparation of composite samples.

International Standards for sampling bulk material, for example coal, iron ore and crude petroleum, have been published already and are being revised in the respective Technical committees dealing with those materials. These standards have been used for transactions in order to contribute to the facilitation and promotion of world trade in these materials. However, there is non-uniformity in the use of technical terms and in the application of statistical methods in these standards, especially between standards drafted by different Technical committees.

Accordingly, one of the purposes of this part of ISO 11648 is to provide a set of technical terms and definitions necessary for sampling from bulk materials in order to give a basis for greater uniformity of technical terms and definitions in future versions of the above-published International Standards and in new standards for other commodities.

Another purpose of this part of ISO 11648 is to give guidance on the application of statistical methods. For example, different methods of bias testing are specified in the above International Standards and the users of them may not be able to judge which is better. This part of ISO 11648 attempts to provide an alternative test method for bias. The mathematical model for the aforementioned test methods cannot be physically implemented with the majority of mechanical sampling systems in existence today. Where the test method can be implemented it does not accurately simulate normal physical operating conditions unless the sampling system is designed to operate that way during normal operations. The proposed test method is an extension of the usual bias test method involving paired data. The test method introduces direct estimation of error variances by means of duplicate measurements of each member of paired data. This provides greater accumulation of knowledge about error variances than any of the methods ever proposed for bias testing.

Furthermore, it has been suggested recently that serial data analysis, such as the variogram method, should be incorporated into sampling plans for bulk materials. This part of ISO 11648 gives information through several applications of serial data analysis to the various kinds of data rather than a standard, because the technique is still in the development stage.

The main purpose of sampling from a commodity of bulk material is for the commerce and trade. Sampling from a commodity is classified into two different procedural types; one is sampling of bulk materials for the accurate estimation of an average value of the quality characteristic assessed in the lot and the other is an inspection procedure for bulk materials for making a decision concerning lot acceptance. International Standards for the first type of procedure are applicable to the sampling of coal, iron ore and other commodities, as is ISO 11648 (all parts). This part is the general introduction of ISO 11648. An International Standard for the second type is ISO 10725.

Sampling of bulk materials can be classified into two categories depending on the field of application; one is sampling from a commodity as described above and the other is sampling in a plant. The purpose of sampling in a plant is to control the production process and to assure the quality of products for users, using data obtained by measurements on the test sample. For example, in operations of a basic oxygen steel-making furnace, samples are taken from the molten steel in order to control mainly the production processes and the results are used to assure that the chemical composition meets the requirements for the product being made. Therefore, methods of sampling in a plant should be managed by the plant itself, but should follow correct sampling procedures as described in the various parts of ISO 11648.

5 Particular problems for sampling bulk materials

When a lot consists of hundreds of bulbs or bolts, random selection of bulbs or bolts gives a representative sample of the lot. In the case of sampling bulk materials, increments are taken from a lot instead of individual bulbs or bolts. In bulk sampling, it is essential to determine the minimum mass of increment.

An example of a sequence of sampling plan decisions involving bulk materials packed in 50 kg sacks (e.g. flour or cement) is:

- select the sacks to sample;
- determine the mass of increment;
- take the increments from the sacks selected with a sampling device that will give a representative sample (i.e. avoiding bias due to stratified layers of product with different properties in the sack);
- perform the necessary sample preparation and tests.

In selecting a sampling device, the points to consider are that too small a device could introduce bias by dropping the larger particles in the lot, while too large a device could result in excessive loads for preparation of the sample. Accordingly, the dimension of the sampling device should be determined by a compromise between these upper and lower device sizes.

However, in the sampling of powder materials, consideration should also be given to the effect of environment and the convenience of handling increments, since the mass of increment calculated using the formula below could be too small to handle easily.

In practice, both manual methods and mechanical methods are usually applied. In the case of sampling particulate materials, the minimum mass of increment for manual sampling is based on the implementation of the dimensions $(3 \times 3 \times 3)\,d$, where d is the nominal top size, expressed in millimetres, of the particles in a lot. The manual increment mass is based on an assumption of random sampling of an increment from a lot.

In sampling from a stopped belt, place a suitably profiled sampling frame, with minimum internal dimensions of three times the nominal top size of the lot or 30 mm, whichever is the larger, on the stationary belt and insert it through the material so that it is in close contact with the belt across its full width. Remove the material within the sampling frame, ensuring that all particles in this area are included in the increment by sweeping the belt, and deposit each increment into a suitable container. Stopped-belt sampling, although not always practical, is a method preferred to other sampling procedures with which it is compared.

The minimum mass of an increment, taken by a cutter-type sampler from the material at the discharge end of a moving stream, is determined by the minimum cutter aperture and the maximum cutter speed. The maximum cutter speed is restricted to avoid bias due to deflection of the larger particles. The increment mass by a cutter-type sampler is usually 10 to 50 times the increment mass by manual sampling. Cross-belt cutters collect the increment from the material stream while it is being conveyed on a conveyor belt. The cutter should cut the bulk material stream in a plane normal to the surface of the conveyor.

In sampling from discrete material, the expected variance of the estimate of the average value of the quality characteristic assessed in the lot is expressed by the following equation:

$$\sigma_{\mathrm{E}}^{2} = \left(1 - \frac{n_{\mathrm{ite}}}{N_{\mathrm{ite}}}\right) \frac{s_{\mathrm{ws}}^{2}}{n_{\mathrm{ite}}} \tag{1}$$

where

N_{ite} is the number of items in a population;

n_{ite} is the number of items in a sample;

s_{ws}^{2} is the variance between items within a sample calculated from the quality characteristic assessed.

In Equation (1), $(1 - n_{\mathrm{ite}}/N_{\mathrm{ite}})$ is called the "finite population correction". If the value of $n_{\mathrm{ite}}/N_{\mathrm{ite}}$ is less than 1/10, then the correction can be omitted. In sampling from bulk material, the value corresponding to $n_{\mathrm{ite}}/N_{\mathrm{ite}}$, i.e. $n_{\mathrm{sub}}/N_{\mathrm{sub}}$, is less than 1/10 in most cases and the finite population correction can be omitted, where n_{sub} is the number of increments taken from a sub-lot and N_{sub} is the total number of possible increments in a sub-lot. This inference is applicable not only to the sampling stage (taking increments) but also to the sample preparation stage (extraction of test sample from a gross sample) and to the analysis stage (taking test portion from a test sample). It is also applicable to liquids and gases. The finite population correction has to be applied to sampling wagons from a train, drums from a truck, etc. in sampling from bulk materials.

The quality characteristics which are to be inspected are usually specified in the transactions. In general, moisture content is determined in order to calculate the dried mass of a lot from the measured wet mass of the lot. Various kinds of chemical compositions, especially representative composition, in dry basis are analysed. In order to calculate the net mass of the representative component, it is important that the weighing precision be balanced for the wet mass of the lot, the moisture content and the representative composition. Particle size distribution and other physical and chemical properties are sometimes determined. Sampling procedures should be established to satisfy all the requirements of each quality characteristic separately.

6 Differences between particulates, liquids and gases

The process of sampling of particulate materials is usually divided into three stages:

a) the process of taking increments,

b) the process of sample preparation, and

c) the process of measurement.

Each process has its own variance component:

- the sampling variance component caused during increment sampling,
- the sample preparation variance component created during test sample preparation, and
- the measurement variance component characterizing the precision of the measurement method (analytical method) used.

If n increments are taken using mass-basis systematic sampling from a lot of particulate materials, a gross sample is composed of n increments, a test sample is prepared from the gross sample and n_{M} measurements are obtained on the test sample, then the variance of estimate of the average value of the quality characteristic assessed, σ_{E}^2, in the lot can be approximated by Equation (2):

$$\sigma_{\mathrm{E}}^2 = \frac{\sigma_{\mathrm{wst}}^2}{n} + \sigma_{\mathrm{P}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_{\mathrm{M}}} \qquad (2)$$

where

σ_{wst}^2 is the variance component between increments within strata including each increment in the lot;

σ_{P}^2 is the variance component between test samples prepared from the gross sample;

σ_{M}^2 is the variance component between measurements obtained on the test sample;

n is the number of increments taken from the lot;

n_{M} is the number of measurements on the test sample.

NOTE The theory of systematic sampling is given in references [1] and [2] of the Bibliography.

If σ_E^2 is required to be less than a limiting value, the second term in Equation (2), σ_P^2, will remain unchanged, whereas the first and the third terms can be reduced by selection of an appropriate combination of number of increments, n, and number of measurements, n_M.

When the variance component between the test samples, σ_P^2, represents the major part of σ_E^2 in Equation (2) and σ_E^2 is required to be less than a limiting value, then a sufficient reduction in σ_E^2 may not be possible by increasing n and n_M. In particular, improvement of the variance component between test samples (variance component of sample preparation) is hard to achieve in the preparation process of particulate materials, due to its nature. The only solution is the subdivision of the lot into an appropriate number of sub lots.

If a lot is subdivided into u_{lot} sub-lots of equal quantity, n_{sub} increments are taken from each sub-lot, a gross sample is constituted for each sub-lot and n_M replicate measurements are obtained on each gross sample, then the variance of the estimate of the average value of the quality characteristic assessed in the lot will be expressed by Equation (3):

$$\sigma_E^2 = \frac{1}{u_{lot}} \left(\frac{\sigma_{wst}^2}{n_{sub}} + \sigma_P^2 + \frac{\sigma_M^2}{n_M} \right) \tag{3}$$

Thus the variance of estimate of average value of the quality characteristic assessed in the lot, σ_E^2, can be adjusted by selecting an appropriate number of sub-lots, u_{lot}. A sub-lot is to be a known quantity of bulk material, in order to calculate the quality of the lot by weighted averaging.

In the process of sampling of liquids, the variation within a gross sample is comparatively small and the process of sample preparation is usually omitted. If necessary, the gross sample may be stirred to make this variation negligible.

In the process of sampling of gases, an increment taken from a lot is subjected directly to analysis and the process of sample preparation is usually omitted.

In the sampling of particulate materials, where possible, all the produced material should preferably be homogenized, possibly including several lots before the increments are taken. Bedding systems for particulate materials are stockpiled before being loaded to vessels so as to reduce the quality variation within the lot. Taking increments from strata, into which a lot is subdivided for smaller variation, also reduces the quality variation. At the sample preparation stage, particle size reduction is another step in the homogenization. At the test sample stage, mechanical mixing is carried out in a laboratory. However, special operations of homogenization at this stage can sometimes lead to segregation of properties.

7 Experimental methods for obtaining variance components at various stages of sampling

7.1 Variance components at various stages of sampling

A bulk-sampling plan, which is to be used in routine sampling, should be established so that a specified overall precision for a lot is obtained taking into account past experience and the results from specially run experiments.

Variance components in routine sampling are usually divided into variance components of sampling (taking increments), sample preparation and measurement. In order to estimate these variance components separately or jointly, the following three types of experiments are used:

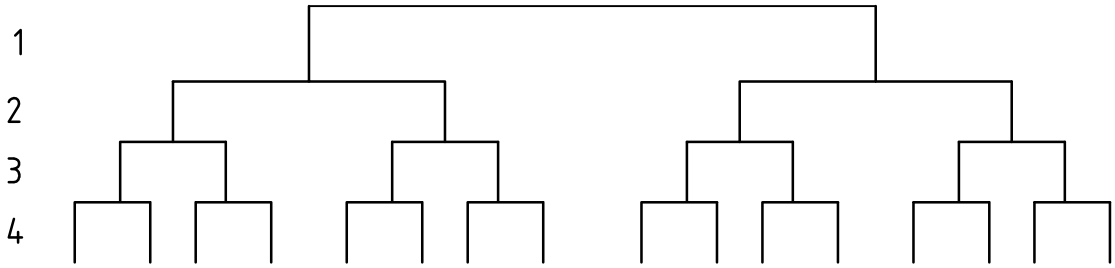
- nested experiments;
- interpenetrating sampling; and
- mass-basis systematic sampling with increment-by-increment measurement.

7.2 Nested experiments

In a completely new sampling situation, where there is no previous experience, a sampling experiment should be done to estimate the variance components at various stages of sampling, i.e. the between-lots variance component, the between-increment variance component, the between-samples variance component and the variance component due to measurement error. The simplest experimental design is a fully nested experiment with two samples or measurements at each stage as shown in Figure 1.

To obtain sufficient information about the variance components between the sampling stages, samples from approximately 20 lots should be tested (although in most situations several pairs of sampling stage samples could be taken from one lot).

The disadvantage is that, for each sampling stage sample, four measurements are needed in the plan shown and this is more than required. The degrees of freedom and the expected mean squares for this example are as shown in Table 3.



Key

1 Lots
2 Sampling stage
3 Sample preparation stage
4 Measurements

Figure 1 — Fully nested experiment

Table 3 — ANOVA with expected mean squares of fully nested experiment

Source	Degrees of freedom	Expected mean square
Between lots	$p-1$	$\sigma_{\mathrm{M}}^2 + 2\sigma_{\mathrm{P}}^2 + 4\sigma_{\mathrm{S}}^2 + 8\sigma_{\mathrm{BL}}^2$
Sampling stage within lots	p	$\sigma_{\mathrm{M}}^2 + 2\sigma_{\mathrm{P}}^2 + 4\sigma_{\mathrm{S}}^2$
Sample preparation stage within sampling stage	$2p$	$\sigma_{\mathrm{M}}^2 + 2\sigma_{\mathrm{P}}^2$
Measurement within sample preparation stage	$4p$	σ_{M}^2
Total	$8p-1$	

σ_{BL}^2 is the variance component between lots;

σ_{S}^2 is the variance component of sampling stage;

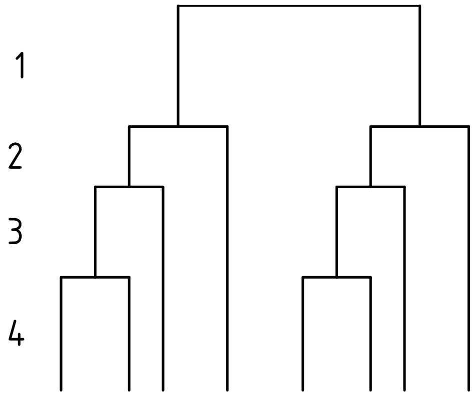
σ_{P}^2 is the variance component of sample preparation stage;

σ_{M}^2 is the variance component of measurement;

p is the number of lots.

The $4p$ degrees of freedom for the measurement variance component are more than needed and a design which distributes the degrees of freedom more evenly would be better.

This can be done using a staggered-nested experiment design as shown in Figure 2.



Key

1 Lots

2 Sampling stage

3 Sample preparation stage

4 Measurements

Figure 2 — Staggered-nested experiment

This cuts down the number of measurements from $8p$ to $4p$ and the degrees of freedom and the expected mean squares are as shown in Table 4.

Table 4 — ANOVA with expected mean squares of staggered-nested experiment

Source	Degrees of freedom	Expected mean square
Between lots	$p-1$	$\sigma_{\mathrm{M}}^2 + (3/2)\sigma_{\mathrm{P}}^2 + (5/2)\sigma_{\mathrm{S}}^2 + 4\sigma_{\mathrm{BL}}^2$
Sampling stage within lots	p	$\sigma_{\mathrm{M}}^2 + (7/6)\sigma_{\mathrm{P}}^2 + (3/2)\sigma_{\mathrm{S}}^2$
Sample preparation stage within sampling stage	p	$\sigma_{\mathrm{M}}^2 + (4/3)\sigma_{\mathrm{P}}^2$
Measurement within second stage	p	σ_{M}^2
Total	$4p-1$	

In these experiments, variance components, σ_{BL}^2, σ_{S}^2, σ_{P}^2 and σ_{M}^2 are estimated, equating each formula in the column of expected mean square to the corresponding observed variance. For details of the fully nested experiment and the staggered-nested experiment, refer to ISO 5725-3. Examples of nested experiments are given in Annex B.

7.3 Interpenetrating sampling

Interpenetrating sampling is applied where the sampling variance component is dominant in comparison with the variance components of sample preparation and measurement. In addition, this is applied where aggregation or accumulation of increments is allowable, i.e. to materials of a particulate or liquid nature.

In mass-basis systematic sampling of iron ore, quality variations within strata including two increments are surveyed periodically. A lot is divided into more than ten parts and even numbered increments are allotted to each part, dividing the number of increments determined according to a mass of lot by the number of parts. Increments are taken at fixed intervals in mass. Odd numbered increments taken from each part and even numbered increments taken from each part are constituted into two composite samples, respectively. (In the following example, these composite samples are denoted by A_i and B_i, respectively, where i is the number of the part). The quality characteristics to be assessed are determined for each composite sample and the quality variations within strata including two increments are estimated.

The methods to be applied are illustrated by the following examples:

EXAMPLE 1

(number of increments per composite sample) × (number of composite samples per part) × (number of parts) = 3 × 2 × 10

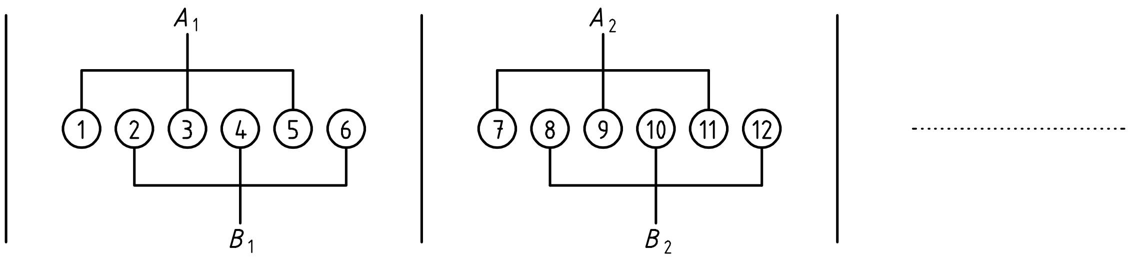


Figure 3 — Interpenetrating sampling

An example carried out on the total iron content is shown in Table 5. In this example, 60 increments are taken from the lot. No. 1, No 3 and No. 5 increments are constituted into composite sample A_1, and No. 2, No. 4 and No. 6 increments are constituted into composite sample B_1. Thus, composite samples A_1 to A_{10} and B_1 to B_{10} are obtained and the total iron content is determined for each composite sample, after preparation of each separately. The range between a_i and b_i is denoted by R_i. From the average range, 0,23, the quality variation within strata including two increments (including also variance components of sample preparation and measurement) is estimated by the following formula:

$$\sigma_{\mathrm{wst}}^2 = 3 \times \left(\frac{\overline{R}}{d_2} \right)^2 \tag{4}$$

where

d_2 is the factor to estimate variance from the range with normal distribution for paired data and equals 1,128;

3 is the number of increments in each composite sample.

Other examples of interpenetrating sampling are shown in C.7.

Table 5 — An example of interpenetrating sampling

Total iron content				
No. of part	*A*	*B*	**Average**	**Range**
1	65,37	64,36	64,865	1,01
2	64,82	64,82	64,82	0
3	64,81	65,10	64,955	0,29
4	64,96	65,06	65,01	0,10
5	65,23	65,20	65,215	0,03
6	65,34	65,22	65,28	0,12
7	65,54	65,80	65,67	0,26
8	65,41	65,34	65,375	0,07
9	65,16	65,22	65,19	0,06
10	65,34	65,69	65,515	0,35
Average			65,19	0,23
σ_{wst}				0,35
Method: 3 × 2 × 10 Steel mill G, 1985-05-19, Tonnage: 97 101 t				

7.4 Mass-basis systematic sampling with increment by increment measurement

Systematic sampling is frequently applied to take increments from bulk materials during transfer instead of simple random sampling from bulk materials in a stationary state because of it is easier to perform and to mechanize. Take increments by systematic sampling and prepare test samples from increments separately and then measure a quality characteristic on each test sample. Data obtained by this way are analysed by the variogram or correlogram method. Data obtained by systematic sampling in mass basis are usually used for this purpose.

The variogram is a plot of the variance as a function of the interval between original data. The distance between consecutive data is called lag one, that between every second data value is called lag two, etc. The value of the variance $V_{\text{exp}}(t)$ corresponding to a lag of k increments can be calculated from the following equation:

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

where

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

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

t lag value for calculating the variogram either on a time or mass basis.

The correlogram is a plot of the coefficient of correlation as a function of the interval between original data. The value of the coefficient of correlation $r_{\text{exp}}(t)$ corresponding to a lag of k increments can be calculated from the following equation:

$$r_{\text{exp}}(t) = \frac{\sum_{i=1}^{n-k} \left(x_i - \overline{x}_i \right)\left(x_{i+k} - \overline{x}_{i+k} \right)}{\sqrt{\sum_{i=1}^{n-k} \left(x_i - \overline{x}_i \right)^2 \sum_{i=1}^{n-k} \left(x_{i+k} - \overline{x}_{i+k} \right)^2}} \tag{6}$$

The variogram and correlogram for a given series have a relationship to one other as described in detail in C.3. One of them or both are applied according to the situations.

Iron ore sampling at a discharging port is usually performed by mechanical equipment with the number of increments determined using systematic sampling on a mass basis with respect to the mass of the lot falling in a stream onto the main belt going from the vessel to a stockpile area.

Increment samples for size analysis are usually sieved increment by increment by a mechanical sieving system for lumpy iron ore. Recently in the sampling of iron ores, increment samples for moisture determination are often measured increment by increment, after preparation if necessary, to avoid moisture loss during storage.

Masses of increments taken by the systematic sampling on a time basis are proportional to the flow rate of the material and the corresponding masses of the lot cannot be known. Accordingly, a quality characteristic of an increment taken on a time basis should not be measured ensuring uniform flow rate.

Thus, serial data including sample preparation errors and measurement errors are provided from routine work. Statistical analysis of serial data is illustrated in Annex C.

The variogram method was primarily developed for obtaining the sampling variance components for each of the sampling plans for several different sampling intervals, e.g. with the sampling interval increased by a factor of two. The variogram value at lag one corresponds to the quality variation within strata including two neighbouring increments in interpenetrating sampling.

However, the variogram method has been used recently for the presentation of special features of serial data, rather than the direct estimation of the sampling variance components.

8 Adjusting the sampling plan to obtain desired precision

In sampling particulate materials, where a lot is subdivided into u_{lot} sub-lots, n_{sub} increments are taken from each sub-lot, a gross sample is constituted for each sub-lot and n_{M} replicate measurements are obtained on each gross sample, then the variance of estimate of average value of the quality characteristic assessed in the lot is expressed by Equation (3):

$$\sigma_{\mathrm{E}}^2 = \frac{1}{u_{\mathrm{lot}}}\left(\frac{\sigma_{\mathrm{wst}}^2}{n_{\mathrm{sub}}} + \sigma_{\mathrm{P}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_{\mathrm{M}}}\right) \tag{3}$$

Here, σ_{E}^2 is given as the desired precision. σ_{wst}^2 and σ_{P}^2 are given according to the material to be sampled. σ_{M}^2 is also given according to the measurement method to be applied to the quality characteristic in question. However, in most cases, σ_{M}^2 is small enough when compared with σ_{wst}^2 and σ_{P}^2. Accordingly, u_{lot} and n_{sub} should be the main parameters to be adjusted in sampling of particulate materials.

In most cases when sampling liquids, the variance component between the test samples prepared from the gross sample, σ_{P}^2, is considered small as it comes from only stirring the gross sample. Accordingly, the variance of estimate of average value of the quality characteristic assessed in the lot will be expressed by Equation (7):

$$\sigma_{\mathrm{E}}^2 = \frac{\sigma_{\mathrm{wl}}^2}{n} + \frac{\sigma_{\mathrm{M}}^2}{n_{\mathrm{M}}} \tag{7}$$

where

σ_{wl}^2 is the variance component between the increments within the lot;

n is the number of increments taken from the lot.

In this case, adjustment is limited to n and n_{M}.

However, when the lot is subdivided into u_{lot} containers of equal mass (sub-lots), the variance of estimate of average value of the quality characteristic assessed in the lot will be expressed by Equation (8):

$$\sigma_{\mathrm{E}}^2 = \frac{1}{u_{\mathrm{lot}}}\left(\frac{\sigma_{\mathrm{wsl}}^2}{n_{\mathrm{sub}}} + \frac{\sigma_{\mathrm{M}}^2}{n_{\mathrm{M}}}\right) \tag{8}$$

where

σ_{wsl}^2 is the variance component between the increments within the container.

In sampling of gases, the accumulation of increments is not considered practical because of its difficulty. Accordingly, the variance of estimate of average value of the quality characteristic assessed in the lot will be expressed by Equation (9):

$$\sigma_{\mathrm{E}}^2 = \frac{1}{n}\left(\sigma_{\mathrm{wl}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_{\mathrm{M}}}\right) \tag{9}$$

where σ_{wl}^2 is the variance component between the increments within the lot.

In this case, n and n_{M} are adjustable.

9 Estimating precision

The precision performed through the routine sampling, sample preparation and measurement procedures should be checked periodically by duplicate sampling.

In the experiments of systematic sampling, twice the number of increments in the routine sampling should be taken at the half interval of the routine sampling and two composite samples, each constituted by n increments respectively, should be aggregated in rotation. Two composite samples per lot should be prepared and measured separately according to the routine procedures. It is preferable that experiments for no less than twenty lots of the same material should be carried out.

Irrespective of the number of duplicate data, a control chart for range as described in ISO 8258 can be applied for detection of out-of-control points and for estimation of the precision performed.

Practical applications are given in Annex D of this part of ISO 11648.

10 Checking for bias

Data obtained by routine sampling are usually used for the calculation of the monetary value of the commodity. Biased data give a biased monetary value. Bias is of importance for both parties concerned, purchaser and supplier.

Bias is a result of the sum of all bias-creating effects of various components in the whole sampling system, from taking increments to measuring a quality characteristic.

Bias will be introduced by the deviations from the design criteria and normal operations of various components in the sampling system. In order to avoid bias, individual components in the sampling system should be checked by comparing them with the design criteria. For example, a cutter in a particulate sampling system should obtain a complete cross-section of the trajectory of a falling stream of particulate materials. When a cutter does not obtain a complete cross-section of the material on the belt (e.g. spoon sampler), bias will obviously be introduced, even though evidence of bias cannot be detected. Details of design criteria relevant to sampling systems for particulate materials is given in ISO 11648-2.

Bias is defined as “the difference between the expectation of the test result and an accepted reference value”. However, in practice, an accepted reference value is unknown. Where an “intrinsically unbiased method” is available in place of an “accepted reference method”, bias is usually discussed in comparison with the test results and the value obtained by the intrinsically unbiased method, as an auxiliary measure. For mechanical sampling from falling streams of particulate materials, an example of an inherently unbiased method could be a stopped belt sampling method applied to the same material.

Let the values of measurements on duplicate increments obtained by a mechanical sampler be denoted by x_{i1} and x_{i2}, and the values of measurements on duplicate increments obtained by stopped belt sampling, y_{i1} and y_{i2}, respectively. Increments of the same sets should be taken as closely together as possible. k is the number of sets of increments, preferably more than twenty.

Calculate:

$$g_i = x_{i1} - x_{i2},\ i = 1, 2, \ldots, k \tag{10}$$

$$s_e^2\ (x) = \frac{\sum_{i=1}^{k} g_i^2}{2k} \tag{11}$$

$$h_i = y_{i1} - y_{i2} \tag{12}$$

$$s_e^2 (y) = \frac{\sum_{i=1}^{k} h_i^2}{2k} \tag{13}$$

Put the larger of $s_e^2(x)$, $s_e^2(y)$ in the numerator of F_0 and calculate the ratio F_0 of the error variances:

$$F_0 = s_e^2(x) / s_e^2(y) \text{ or } F_0 = s_e^2(y) / s_e^2(x) \tag{14}$$

If $F_0 > F_{\alpha/2}(v_1, v_2)$, then the null hypothesis, $s_e^2(x) = s_e^2(y)$, is rejected, and the two groups of data cannot be assumed to be drawn from populations with a common variance. The significance level α is usually set equal to 0,05, and v_1 and v_2 are the number of degrees of freedom of $s_e^2(x)$ and $s_e^2(y)$, respectively, and both are k in this case.

If $F_0 < F_{\alpha/2}(v_1, v_2)$, the two groups of data may be assumed to have a common variance.

95 % confidence limits, $T_1(x)$, $T_2(x)$ and $T_1(y)$, $T_2(y)$ are calculated as follows:

$$T_1(x), T_2(x) = \overline{\overline{x}} \pm t_{1-\alpha/2}(k) s_e(x) \tag{15}$$

$$T_1(y), T_2(y) = \overline{\overline{y}} \pm t_{1-\alpha/2}(k) s_e(y) \tag{16}$$

where

$\overline{\overline{x}}$ is the grand average of x_{i1} and x_{i2};

$\overline{\overline{y}}$ is the grand average of y_{i1} and y_{i2}.

Calculate:

$$d_i = \frac{x_{i1} + x_{i2}}{2} - \frac{y_{i1} + y_{i2}}{2} \tag{17}$$

$$s_d^2 = \frac{1}{k-1}\left[\sum_{i=1}^{k} d_i^2 - \frac{1}{k}\left(\sum_{i=1}^{k} d_i\right)^2\right] \tag{18}$$

$$A_2 = \left[t_{1-\alpha/2}(\nu)/\sqrt{k}\right]s_d \tag{19}$$

If $\left|\,\bar{d}\,\right| > A_2$, then the hypothesis of the equality of two means is rejected. The estimate of bias is $\bar{d}$.

If the absolute value of $\bar{d}$ is larger than the maximum tolerable bias, δ, removal of the bias should be considered from the point of view of the actual effects of the bias on the evaluation of the lot.

As for statistical methods for bias testing, various approaches have been proposed in many International Standards in respective fields. However, the method to be applied should be evaluated with regard to availability and efficiency. The method proposed here will give a basic approach for bias testing and an accumulation of knowledge about random errors relating to the material dealt with and the measurement method applied. Detailed discussions through practical applications will be given in Annex E.

11 Precision and bias at measurement stage

Precision and bias at the measurement stage should be reviewed in accordance with all parts of ISO 5725, together with the methods given in this part of ISO 11648.

Annex A
(informative)

Explanatory notes on definitions

A.1 General

This annex gives a necessary explanation of the terms selected in order to avoid confusion or misusage of these terms.

A.2 Bulk material

"Bulk sampling" is defined in 4.27 of ISO 3534-1:1993, while "bulk material" is not defined in ISO 3534 (all parts). However, the definition of "bulk material" will be given in the future version of ISO 3534, as shown in 3.1.1.

Bulk material covers all kinds of materials in which increments are not initially distinguishable, such as particulate material, liquids and gases. This also covers peculiar bulk materials such as cotton and iron scrap. The principles of sampling, such as random drawing of samples at random and stratification of the lot, may be also applied to peculiar bulk materials. However, special consideration should be given to the taking of increment(s) from the materials.

A.3 Sample

The same definition of sample is given in 4.2 of ISO 3534-1:1993 and in 2.1.1 of ISO 3534-2:1993 as "one or more sampling units taken from a population and intended to provide information on the population" with a note "a sample may serve as a basis for a decision on the population or on the process which produced it". In the future version of ISO 3534, the term will be defined as "subset of a specified population made up of one or more sampling units".

A.4 Sampling

The same definition of "sampling" is given in both 4.4 of ISO 3534-1: 1993 and 2.2 of ISO 3534-2:1993. The slightly modified definition will be given in the future version of ISO 3534 as shown in 3.1.3. Fundamental to the accurate estimation of an average value of the quality characteristic assessed in the lot is the taking of a simple random sample from a lot. However, simple random sampling is a difficult procedure, in particular from a lot in stationary state (static sampling).

Instead of simple random sampling from a lot in a stationary state, systematic sampling in time or in mass is applied during transfer of a lot for easy ease of execution (dynamic sampling).

Multi-stage sampling is sometimes applied according to the form of a lot, such as a train comprising a number of wagons.

An appropriate procedure for implementing these sampling plans can be established on the basis of knowledge about the quality variation in a lot, the variance component of sample preparation and the variance component of measurement. The quality variation is determined from the results of experimental sampling, such as interpenetrating sampling. The variance component of sample preparation and the variance component of measurement are obtained by a suitably designed experiment.

Precision attained by routine sampling is verified by check sampling, such as duplicate sampling. The bias of routine sampling cannot be determined in general. Increments taken by a mechanical sampler can be compared with increments taken from the corresponding point of the conveyor belt during stoppage. Individual components in the sample preparation process, such as dividers in a mechanical system, can also be checked for bias by an appropriate experiment.

A.5 Lot

The term “consignment” is defined in 1.3.7 of ISO 3534-2:1993 and has been used instead of “lot” in the Standards drafted by some technical committees. However, more recently, the term “lot” is usually used for sampling of bulk material. The term “lot” can be found in the Standards published in 1994 (see ISO 9411-1). On the other hand, “lot ⟨inspection⟩” is defined in 1.3.5 of ISO 3534-2:1993. In order to distinguish from this, “lot ⟨bulk sampling⟩” is newly defined.

A.6 Sub-lot, sampling unit and increment

The term “sub-lot” is not defined in ISO 3534:1993, but should be introduced to be confined to the field of bulk sampling in future version of ISO 3534 describing the subdivision of a lot in order to obtain a desired precision, as described in Clause 6.

The term “sampling unit” is defined in 4.1 of ISO 3534-1:1993 and 1.3.3 of ISO 3534-2:1993 wholly in the same wordings having two meanings with two notes. In the first definition, the term is defined as “one of the individual units into which a population is divided”. While, the second definition is that “a quantity of product, material or service forming a cohesive entity and taken from one place and at one time to form a part of a sample”. In the future version of ISO 3534, the definition will be given in this form for easier understanding for users.

The term “increment” is defined in 4.25 of ISO 3534-1:1993, as “a sampling unit in the case of bulk sampling; i.e. a quantity of material taken at one time by one action from a larger body of material”. However, in the future version of ISO 3534, the same definition as defined in this International Standard that “quantity of bulk material taken in one action by a sampling device” will be given.

In order to understand the mutual relationship between these terms, consider the following sampling practice in loading coal into a vessel.

Suppose 70 000 t of coal in wagons are loaded onto a vessel directly, though in fact the main part of the coal to be loaded is usually supplied from coal stocked in a pile and only a small part of the coal to be loaded is supplied by a train directly. On each wagon, 100 t of coal are loaded. A train is made up of a hundred wagons and 70 000 t of coal are delivered by seven trains to the loading facilities. Sampling equipment is situated so as to intercept the falling stream at the transfer head of the conveyor belt subsequent to a tipple. A weighing machine is installed on the conveyor belt after the tipple so that the mass of coal passed through the location of the sampling equipment can be measured by an appropriate time lag correction.

Case 1: Routine sampling is carried out by the sampling equipment. At 500 t intervals, n (e.g. 140) increments are taken according to the indications of the weighing machine and 20 increments representative of each 10 000 t sub-lot are composed to make a gross sample and seven gross samples are combined successively to represent each train. These gross samples are prepared into seven test samples separately and these test samples are analysed separately. The average value of the quality characteristic assessed in the lot is determined by averaging these seven test results.

Case 2: Routine sampling cannot be carried out due to an unfortunate breakdown of the sampling equipment. As agreed between the parties concerned with delivery, an alternative sampling procedure is carried out using an auger sampler from the wagons before the tipple. Ten wagons are selected at random from 100 wagons in each train. Two increments are taken from the wagons selected and seven composite samples are composed to represent each train. Composite samples are prepared separately and test samples are analysed separately. The average value of the quality characteristic assessed in the lot is determined by averaging the seven test results.

Case 3: Under the same circumstances as Case 2 above, sampling may be carried out by reducing the number of increments (e.g. 40 per lot) in accordance with an agreement between the parties concerned. Four trains are selected at random from seven trains and five wagons are selected at random from 100 wagons which make up the train selected. Two increments per selected wagon, 40 increments in total, are taken and four composite samples are composed to represent each train selected. Composite samples are prepared separately and test samples are analysed separately. The average value of the quality characteristic assessed in the lot is determined by averaging the four test results.

An example of mass-basis systematic sampling is shown in Case 1. Selection of wagons in Case 2 is an example of stratified sampling, where the strata are trains. In Case 3, an example of three-stage sampling is shown, where four trains are selected as the primary sampling units at the first stage, five trains are selected as the secondary sampling units from the selected train at the second stage and two increments are taken from the selected wagons as the tertiary sampling unit.

The variance of estimates of average value of the quality characteristic assessed in the lot in cases 1, 2 and 3 are expressed by Equations (A.1), (A.2) and (A.3), respectively:

$$\sigma_{\mathrm{E}}^2 = \frac{1}{7}\left(\frac{\sigma_{\mathrm{wst}}^2}{20} + \sigma_{\mathrm{P}}^2 + \sigma_{\mathrm{M}}^2\right) \tag{A.1}$$

where σ_{wst}^2 is the variance component within stratum.

$$\sigma_{\mathrm{E}}^2 = \frac{1}{7}\left(\frac{\sigma_{\mathrm{BW}}^2}{10} + \frac{\sigma_{\mathrm{wst}}^2}{10 \times 2} + \sigma_{\mathrm{P}}^2 + \sigma_{\mathrm{M}}^2\right) \tag{A.2}$$

where

σ_{BW}^2 is the variance component between wagons in a train;

σ_{wst}^2 is the variance component within stratum in a wagon.

$$\sigma_{\mathrm{E}}^2 = \frac{3}{7}\left(\frac{\sigma_{\mathrm{BT}}^2}{4}\right) + \frac{1}{4}\left(\frac{\sigma_{\mathrm{wst}}^2}{5 \times 2} + \sigma_{\mathrm{P}}^2 + \sigma_{\mathrm{M}}^2\right) \tag{A.3}$$

where

σ_{BT}^2 is the variance component between trains;

$\frac{3}{7}$ reflects the finite population correction in selection of four trains from seven trains.

In Case 1, the first sub-lot of 10 000 t of coal is conceptually divided from the second one by a mass reading at the given increment. The material in a falling stream is a continuous flow and its parts are not separated from each other. However, each 10 000 t of coal is called a sub-lot. In intermittent sampling, some sub-lots are not selected. Accordingly, a sub-lot can be a primary sampling unit. Increments taken from a sub-lot are secondary sampling units.

In Case 2, each 10 000 t of coal is a sub-lot and at the same time a primary sampling unit. Wagons selected from a train are secondary sampling units. Increments taken from a selected wagon are tertiary sampling units.

In Case 3, trains are primary sampling units. Wagons in a selected train are secondary sampling units. Increments taken from a selected wagon are tertiary sampling units.

The term “sampling unit” is used in the definition of “sample”, “simple random sampling”, “stratified sampling” and “multi-stage sampling”.

A.7 Composite sample

The term “aggregated sample” is defined in 4.28 of ISO 3534-1:1993 with the same meaning as “composite sample” in this International Standard. As “aggregated” is not used in the practice of bulk sampling, the term is replaced by “composite”. “Composite sample” should be used for non-routine sampling such as duplicate sampling for checking precision, interpenetrating sampling for investigation of quality variation and inspection sampling, while “gross sample” is used for routine sampling.

A.8 Gross sample

The term “gross sample” is defined in 4.29 of ISO 3534-1:1993 as the representative sample of a population. As described in Clause 6, sub-division of the lot into sub-lots is necessary for obtaining the designed precision. However, if a lot is small enough, the lot remains undivided. Nevertheless, the mass of one undivided lot would be less than the usual mass of a sub-lot. Accordingly, the gross sample should also be defined as the representative sample for both a lot and a sub-lot. In addition, the term “gross sample” should be confined in usage to routine sampling to avoid confusion with “composite sample”.

A.9 Test sample and test portion

A part of a test sample for chemical analysis (a test portion) is usually used for chemical analysis at one time. For test samples taken for other purposes than chemical analysis, either a part of the test sample or the whole quantity of the test sample is used for the test at one time.

A.10 Routine sampling and routine sample preparation

The procedures of routine sampling and routine sample preparation can be established on the basis of experimental work considering the practical application and are stipulated in the respective International Standard for sampling. The procedures of sampling and sample preparation in experiments should be distinguished from those of routine sampling and routine sample preparation. Routine sampling and routine sample preparation are sometimes accomplished by an integrated sampling-and-sample-preparation system, which is followed by instrumental analysis.

A.11 Sample division

Devices for sample division are classified into two types; one is the increment type and the other is the riffle type. The variance component of sample division by the increment may be estimated theoretically from the variance component between the increments at that stage. Sample division by a riffle is carried out by dividing the particles in a sample into the opposite sides of the edged plate at random. The variance component of sample division by the riffle may also be estimated from the results of the experiment.

However, investigation of variance components at different stages of sample division generally requires laborious experiments. Routine sample preparation procedures as a whole process can be checked by making duplicate tests.

In specific International Standards for sample preparation, the minimum mass of the sample to be retained after division at different stages should be stipulated and should be based on results obtained by experimental investigations so as to attain the required precision of sample preparation.

A.12 Sampling procedure, sample preparation procedure, sampling plan, sampling scheme and sampling system

The terms of (sampling) procedure, (sample preparation) procedure, (sampling) plan, (sampling) scheme and (sampling) system are used frequently in standards on bulk sampling. However, these terms are already defined in 2.3.2, 2.3.3, 2.3.4 and 2.3.5 of ISO 3534-2:1993 for acceptance sampling. Accordingly, the terms have been redefined for bulk sampling so as to avoid confusion with terms given in acceptance sampling standards, such as ISO 10725.

Annex B
(informative)

Fully-nested experiments

B.1 General considerations

When one has no knowledge about variance components of sampling, sample preparation and measurement, nested experiments may be attempted in order to obtain the necessary information about these variance components. Procedures for sampling, sample preparation and measurement can be established as a routine method. The experiment should be carried out on at least 10 lots, but preferably 20 lots. Statistical analyses by control chart and by ANOVA (analysis of variance) are given in the following.

B.2 Experimental parameters

Parameters for the experiment are as follows.

Quality characteristic: ash content (%).

Lot:

- material: coal for coke making;
- transport mode: ship;
- number of lots: 20;
- mass of lots: average 8 345 t; minimum 7 000 t; maximum 9 800 t.

Sampling:

- nominal top size of lot: 50 mm;
- mass of increment: 1,5 kg;
- number of increments taken from one lot: 30 × 2 = 60;
- method of taking increments: stop the conveyor belt that is unloading coal from the ship at the derived interval, determined by dividing the mass of the lot by the number of increments to be taken, and using a shovel take 1,5 kg from the surface material at the correct increment on the belt conveyor.

Sample preparation:

- composite samples: put individual increments taken consecutively, in sample containers A and B alternatively, and regard them as the composite samples A and B consisting of 30 increments;
- test samples: two test samples are prepared from each composite sample.

Measurement:

- ash contents are analysed in duplicate for each test sample.

B.3 Results of experiment

Results of the fully nested experiments described above are shown in Table B.1.

Table B.1 — Results of fully nested experiments

Lot No.	Ash %							
	x_{111}	x_{112}	x_{121}	x_{122}	x_{211}	x_{212}	x_{221}	x_{222}
1	9,38	9,24	9,02	8,98	9,22	9,32	8,40	8,38
2	9,76	9,80	9,92	9,92	9,36	9,36	9,72	9,54
3	7,40	7,26	7,32	7,40	7,55	7,61	7,57	7,49
4	8,62	8,76	8,82	8,84	9,20	9,34	10,00	10,00
5	9,16	9,18	8,72	8,68	8,89	8,75	9,51	9,47
6	9,08	9,08	9,06	8,86	8,80	8,84	8,76	8,60
7	8,77	8,69	8,77	8,75	9,16	8,92	9,06	8,94
8	8,62	8,68	8,80	8,42	8,78	9,02	8,62	8,94
9	8,60	8,74	7,10	7,22	8,88	9,10	9,08	9,00
10	6,96	7,20	7,32	7,40	8,59	8,89	7,55	7,43
11	8,44	8,26	7,92	7,70	8,65	8,45	8,37	8,15
12	8,24	8,00	8,38	8,12	8,42	8,26	8,78	8,72
13	7,21	7,25	6,85	7,03	7,21	7,31	7,31	7,39
14	8,84	9,00	8,96	8,90	9,24	9,16	9,20	9,38
15	8,45	8,51	8,91	8,79	9,00	9,06	8,86	8,96
16	9,02	9,08	9,16	9,08	8,75	8,83	8,65	8,75
17	8,71	8,77	8,75	8,75	8,98	8,96	9,00	9,18
18	8,77	8,92	9,24	9,32	8,82	8,64	8,32	8,42
19	7,37	7,39	7,13	7,25	7,10	6,92	6,64	6,74
20	10,12	10,02	9,96	9,94	10,72	10,78	10,30	10,30

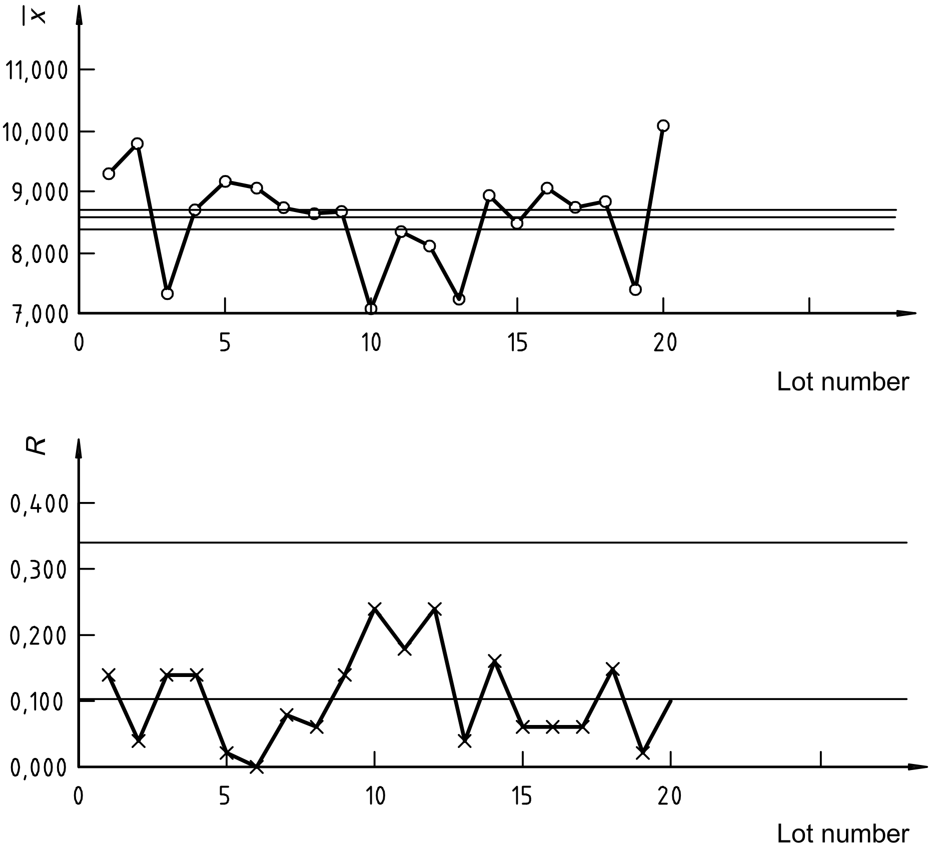
NOTE x_{111}, x_{112}, …, x_{222} are the results corresponding to measurements shown in Figure 1.

B.4 Statistical analysis by control chart

B.4.1 Control chart

A control chart of part of the data at the measurement stage is shown in Figure B.1 as an example.

Similarly, control charts at the test sample stage and at the composite sample stage can be drawn.



Key

$\bar{x}$ = Mean

R = Range

Figure B.1 — Control chart at measurement stage

B.4.2 Interpretation of control charts

A data point on an mean chart is the average of two measurements on a test sample, while a data point on a range chart is the range of two measurements for a test sample.

In the range chart, no out-of-control point is observed at the measurement stage. In the mean chart at the measurement stage in this example, 14 out-of-control points out 20 points are observed. A state of control in the range chart and out-of-control points in the mean chart mean that the precision expressed in the range chart is stable and precise enough to detect the variation expressed by these out-of-control points in the mean chart, contrary to the usual control charts where no out-of-control point is expected to be observed.

B.4.3 Calculation of variance components at each stage

The following values are obtained while making range charts (see also Table B.2):

— $\bar{R}_1$ at the measurement stage;

— $\bar{R}_2$ at the test sample stage;

— $\bar{R}_3$ at the composite sample stage.

At the measurement stage, $\bar{R}_1$ is equal to:

$$\bar{R}_1 = \frac{\left(\sum_{i=1}^{20}\left|x_{111i} - x_{112i}\right| + \sum_{i=1}^{20}\left|x_{121i} - x_{122i}\right| + \sum_{i=1}^{20}\left|x_{211i} - x_{212i}\right| + \sum_{i=1}^{20}\left|x_{221i} - x_{222i}\right|\right)}{80}$$

$$= 0{,}112\ 875$$

At the test sample stage, $\bar{R}_2$ is equal to:

$$\bar{R}_2 = \frac{\left(\sum_{i=1}^{20}\left|x_{11.i} - x_{12.i}\right| + \sum_{i=1}^{20}\left|x_{21.i} - x_{22.i}\right|\right)}{40}$$

$$= 0{,}293\ 755$$

At the composite sample stage, $\bar{R}_3$ is equal to:

$$\bar{R}_3 = \frac{\left(\sum_{i=1}^{20}\left|x_{1..i} - x_{2..i}\right|\right)}{20}$$

$$= 0{,}417\ 625\ 5$$

$$\hat{\sigma}_{\mathrm{M}}^2 = \left(\frac{\bar{R}_1}{d_2}\right)^2 = \left(\frac{0{,}112\ 9}{1{,}128}\right)^2 = 0{,}0100 \qquad \text{(B.1)}$$

$$\hat{\sigma}_{\mathrm{P}}^2 = \left(\frac{\bar{R}_2}{d_2}\right)^2 - \frac{1}{2}\left(\frac{\bar{R}_1}{d_2}\right)^2 = \left(\frac{0{,}294\ 4}{1{,}128}\right)^2 - \frac{1}{2}\left(\frac{0{,}112\ 9}{1{,}128}\right)^2 = 0{,}063\ 1 \qquad \text{(B.2)}$$

$$\hat{\sigma}_{\mathrm{S}}^2 = \left(\frac{\bar{R}_3}{d_2}\right)^2 - \frac{1}{2}\left(\frac{\bar{R}_2}{d_2}\right)^2 = \left(\frac{0{,}417}{1{,}128}\right)^2 - \frac{1}{2}\left(\frac{0{,}294\ 4}{1{,}128}\right)^2 = 0{,}103\ 0 \qquad \text{(B.3)}$$

where

$\hat{\sigma}_{\mathrm{M}}^2$ is the variance component at the measurement stage;

$\hat{\sigma}_{\mathrm{P}}^2$ is the variance component between test samples (variance component of sample preparation);

$\hat{\sigma}_{\mathrm{S}}^2$ is the variance component between composite samples (variance component of sampling);

d_2 is the factor for estimating standard deviation from the range of normally distributed paired data and for n = 2, d_2 = 1,128.

Table B.2 — Calculation of variance components at various stages

Lot No.	Results																					
	x_{111}	x_{112}	$x_{11.}$	R_1	x_{121}	x_{122}	$x_{12.}$	R_1	$x_{1..}$	R_2	x_{211}	x_{212}	$x_{21.}$	R_1	x_{221}	x_{222}	$x_{22.}$	R_1	$x_{2..}$	R_2	$x_{...}$	R_3
1	9,38	9,24	9,31	0,14	9,02	8,98	9	0,04	9,155	0,31	9,22	9,32	9,27	0,1	8,4	8,38	8,39	0,02	8,83	0,88	8,992 5	0,325
2	9,76	9,8	9,78	0,04	9,92	9,92	9,92	0	9,85	0,14	9,36	9,36	9,36	0	9,72	9,54	9,63	0,18	9,495	0,27	9,672 5	0,355
3	7,4	7,26	7,33	0,14	7,32	7,4	7,36	0,08	7,345	0,03	7,55	7,61	7,58	0,06	7,57	7,49	7,53	0,08	7,555	0,05	7,45	0,21
4	8,62	8,76	8,69	0,14	8,82	8,84	8,83	0,02	8,76	0,14	9,2	9,34	9,27	0,14	10	10	10	0	9,635	0,73	9,197 5	0,875
5	9,16	9,18	9,17	0,02	8,72	8,68	8,7	0,04	8,935	0,47	8,89	8,75	8,82	0,14	9,51	9,47	9,49	0,04	9,155	0,67	9,045	0,22
6	9,08	9,08	9,08	0	9,06	8,86	8,96	0,2	9,02	0,12	8,8	8,84	8,82	0,04	8,76	8,6	8,68	0,16	8,75	0,14	8,885	0,27
7	8,77	8,69	8,73	0,08	8,77	8,75	8,76	0,02	8,745	0,03	9,16	8,92	9,04	0,24	9,06	8,94	9	0,12	9,02	0,04	8,882 5	0,275
8	8,62	8,68	8,65	0,06	8,8	8,42	8,61	0,38	8,63	0,04	8,78	9,02	8,9	0,24	8,62	8,94	8,78	0,32	8,84	0,12	8,735	0,21
9	8,6	8,74	8,67	0,14	7,1	7,22	7,16	0,12	7,915	1,51	8,88	9,1	8,99	0,22	9,08	9	9,04	0,08	9,015	0,05	8,465	1,1
10	6,96	7,2	7,08	0,24	7,32	7,4	7,36	0,08	7,22	0,28	8,59	8,89	8,74	0,3	7,55	7,43	7,49	0,12	8,115	1,25	7,667 5	0,895
11	8,44	8,26	8,35	0,18	7,92	7,7	7,81	0,22	8,08	0,54	8,65	8,45	8,55	0,2	8,37	8,15	8,26	0,22	8,405	0,29	8,242 5	0,325
12	8,24	8	8,12	0,24	8,38	8,12	8,25	0,26	8,185	0,13	8,42	8,26	8,34	0,16	8,78	8,72	8,75	0,06	8,545	0,41	8,365	0,36
13	7,21	7,25	7,23	0,04	6,85	7,03	6,94	0,18	7,085	0,29	7,21	7,31	7,26	0,1	7,31	7,39	7,35	0,08	7,305	0,09	7,195	0,22
14	8,84	9	8,92	0,16	8,96	8,9	8,93	0,06	8,925	0,01	9,24	9,16	9,2	0,08	9,2	9,38	9,29	0,18	9,245	0,09	9,085	0,32
15	8,45	8,51	8,48	0,06	8,91	8,79	8,85	0,12	8,665	0,37	9	9,06	9,03	0,06	8,86	8,96	8,91	0,1	8,97	0,12	8,817 5	0,305
16	9,02	9,08	9,05	0,06	9,16	9,08	9,12	0,08	9,085	0,07	8,75	8,83	8,79	0,08	8,65	8,75	8,7	0,1	8,745	0,09	8,915	0,34
17	8,71	8,77	8,74	0,06	8,75	8,75	8,75	0	8,745	0,01	8,98	8,96	8,97	0,02	9	9,18	9,09	0,18	9,03	0,12	8,887 5	0,285
18	8,77	8,92	8,845	0,15	9,24	9,32	9,28	0,08	9,062 5	0,435	8,82	8,64	8,73	0,18	8,32	8,42	8,37	0,1	8,55	0,36	8,806 25	0,512 5
19	7,37	7,39	7,38	0,02	7,13	7,25	7,19	0,12	7,285	0,19	7,1	6,92	7,01	0,18	6,64	6,74	6,69	0,1	6,85	0,32	7,067 5	0,435
20	10,12	10,02	10,07	0,1	9,96	9,94	9,95	0,02	10,01	0,12	10,72	10,78	10,75	0,06	10,3	10,3	10,3	0	10,525	0,45	10,267 5	0,515
Total				2,07				2,12		5,235				2,6				2,24		6,54		8,352 5

Average (R_1) = (2,07+2,12+2,6+2,24)/80 = 0,112 875

Average (R_2) = (5,235+6,54)/40 = 0,294 375

Average (R_3) = 8,352 5/20 = 0,417 625

B.5 Statistical analysis using ANOVA

Results of nested experiments can also be analysed using ANOVA (analysis of variance). The ANOVA table is shown in Table B.3.

Table B.3 — ANOVA of fully nested experiments on ash

Source of variation	Sum of squares	Degrees of freedom	Mean square	Expected mean square
Lots	96,172	19	5,062	$\sigma_M^2 + 2\sigma_P^2 + 4\sigma_S^2 + 8\sigma_{BL}^2$
Composite samples	9,373	20	0,469	$\sigma_M^2 + 2\sigma_P^2 + 4\sigma_S^2$
Test samples	7,679	40	0,192	$\sigma_M^2 + 2\sigma_P^2$
Measurements	0,777	80	0,010	σ_M^2
Total	114,00	159		
σ_{BL}^2 is the variance component between lots.				

In Table B.3, the mean squares are unbiased estimates of parameters estimated, respectively. Consequently:

$$\hat{\sigma}_M^2 + 2\hat{\sigma}_P^2 + 4\hat{\sigma}_S^2 + 8\hat{\sigma}_{BL}^2 = 5{,}062$$

$$\hat{\sigma}_M^2 + 2\hat{\sigma}_P^2 + 4\hat{\sigma}_S^2 = 0{,}469$$

$$\hat{\sigma}_M^2 + 2\hat{\sigma}_P^2 = 0{,}192$$

$$\hat{\sigma}_M^2 = 0{,}010$$

then

$$\hat{\sigma}_M^2 = 0{,}01$$

$$\hat{\sigma}_P^2 = 0{,}09$$

$$\hat{\sigma}_S^2 = 0{,}07$$

$$\hat{\sigma}_{BL}^2 = 0{,}57$$

are obtained.

As for σ_M^2, the variance component at final stage, the estimated obtained from the control chart is exactly equal to the value obtained by ANOVA. However, considerable differences are observed between variance components at intermediate stages, σ_P^2 and σ_S^2 obtained by both methods, respectively. One possible reason would be out-of-control points in range charts (lack of normality). Another possible reason would be an error in estimating the variance component.

Confidence intervals for the variance component can be calculated by the methods of Satterthwaite[5] using the chi-squared distribution, or Anderson-Bancroft[6] or Moriguchi[7] using the F-distribution.

a) Method of Satterthwaite

In the case of a one-factor experiment,

$$\frac{v\hat{\sigma}_{\mathrm{A}}^{2}}{\chi_{2}^{2}\left(v,\frac{\alpha}{2}\right)} < \sigma_{\mathrm{A}}^{2} < \frac{v\hat{\sigma}_{\mathrm{A}}^{2}}{\chi_{1}^{2}\left(v,1-\frac{\alpha}{2}\right)} \tag{B.4}$$

where

$$v = \frac{\left(V_{\mathrm{A}} - V_{\mathrm{E}}\right)^{2}}{V_{\mathrm{A}}^{2}/v_{\mathrm{A}} + V_{\mathrm{E}}^{2}/v_{\mathrm{E}}} = \frac{\left(F_{\mathrm{o}} - 1\right)^{2}}{F_{\mathrm{o}}/v_{\mathrm{A}} + 1/v_{\mathrm{E}}}$$

v_{A} and v_{E} are the number of degrees of freedom for variances V_{A} and V_{E} respectively.

b) Method of Anderson-Bancroft

In the case of a one-factor experiment,

$$\frac{\left(F_{\mathrm{o}}/F_{2}\right)-1}{F_{\mathrm{o}}-1}\hat{\sigma}_{\mathrm{A}}^{2} < \sigma_{\mathrm{A}}^{2} < \frac{\left(F_{\mathrm{o}}/F_{1}\right)}{F_{\mathrm{o}}-1}\hat{\sigma}_{\mathrm{A}}^{2} \tag{B.5}$$

where

$$F_{\mathrm{o}} = V_{\mathrm{A}}/V_{\mathrm{E}}$$

$$F_{2} = F\left(v_{\mathrm{A}},v_{\mathrm{E}};\alpha/2\right)$$

$$F_{1} = 1/F\left(v_{\mathrm{E}},v_{\mathrm{A}};\alpha/2\right)$$

c) Method of Moriguchi

In the case of a one-factor experiment with a one-sided design, with expected variances $E\left(V_{\mathrm{A}}\right) = \sigma_{\mathrm{E}}^{2} + r\sigma_{\mathrm{A}}^{2}$ and $E\left(V_{\mathrm{E}}\right) = \sigma_{\mathrm{E}}^{2}$, a 100(1–$\alpha$) % confidence interval for σ_{A}^{2} is

$$\frac{V_{\mathrm{A}}}{n_{\mathrm{o}}}\left[\frac{1}{F\left(v_{\mathrm{A}},\infty\right)} - \frac{1}{F_{\mathrm{o}}} - b_{\mathrm{L}}\left(\frac{1}{F_{\mathrm{o}}}\right)^{2}\right] < \sigma_{\mathrm{A}}^{2} < \frac{V_{\mathrm{A}}}{n_{\mathrm{o}}}\left[F\left(\infty,v_{\mathrm{A}}\right) - \frac{1}{F_{\mathrm{o}}} + b_{\mathrm{U}}\left(\frac{1}{F_{\mathrm{o}}}\right)^{2}\right] \tag{B.6}$$

where

v_{A} is the number of degrees of freedom of V_{A};

b_{U}, b_{L}, $F(\infty,v_{\mathrm{A}})$ and $1/F(\infty,v_{\mathrm{A}})$ are given in the table of reference [7] of the Bibliography.

In the experiment of duplicate measurements of chemical composition, x %; on 24 bulk lots, the variance component of sampling and between lots $\left(\sigma_S^2 + \sigma_L^2\right)$ have been estimated to be equal to 10,17 separately from variance component of measurement. Three methods give the respective lower and upper confidence limits of 90 % to mean value 10,17 as follows:

	Lower limit	**Upper limit**
Method of Satterthwaite	6,47	18,71
Method of Anderson-Bancroft	4,81	20,35
Method of Moriguchi	6,40	17,98

An example of the application of confidence intervals for variance components to the experimental results of sample division can be found in reference [5] of the Bibliography.

Annex C
(informative)

Statistical analysis of serial data

C.1 General

This annex gives practical applications of the statistical analysis of serial data. Some of the examples of the statistical analysis of serial data may be found in the papers by Jowett and others [8],[9],[10],[11],[12]. References [8] and [9] are applications to coal sampling, while reference [11] is an application to blast furnace data. In the former applications, the variogram method was primarily developed for obtaining the sampling variance components for each of the sampling plans for several different sampling intervals, e.g. with the sampling interval increased by a factor of two. In the latter, types of variations of serial data are mainly discussed. Recently, statistical analysis of serial data is also discussed by Gy relating to the sampling of bulk materials (see references [13] to [15] in the Bibliography).

The fundamental aim of statistical analysis of serial data is to determine the types of variation. Types of variation are usually classified into the typical four patterns:

a) random,

b) cyclic,

c) trend;

d) correlated variations.

In fact, two or three of these patterns can be combined and appear in a combined form within a variogram or a correlogram. When a variogram or a correlogram is flat, the variation of the original series is random. When a variogram or a correlogram is cyclic, the variation of original series is also cyclic with the same period. When a variogram or a correlogram is a quadratic curve, the variation of the original series is either a trend or an autocorrelation. In this case, the correlogram is used conveniently to distinguish between a trend and an autocorrelation, by referring to significant values of the coefficient of correlation.

When the variation of the original series is other than random, special consideration should be given to the selection of the sampling interval used in systematic sampling and to the application of either systematic sampling or systematic random sampling.

This annex gives practical applications experienced during the latest decade relating to:

- blast furnace data;
- thickness of paper;
- moisture content in iron ores;
- size distribution and moisture content in iron ores;
- trends in iron ores.

Applications of serial data analysis to blast furnace data and thickness of paper product are introduced to show the variety of patterns in variograms and correlograms. The fields of application of statistical analysis of serial data will be expanded rapidly in the near future with the assistance of computers.

C.2 Fundamentals

The variogram is a plot of variance as a function of the interval between original data. The distance between consecutive data is called lag one, that between every second data value is called lag two, etc. (see Figure C.1).

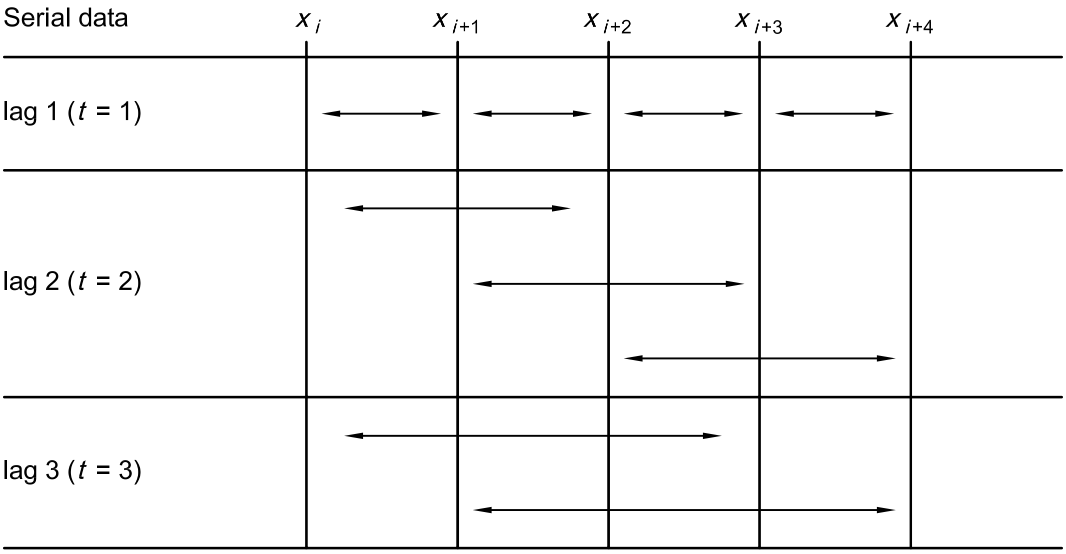


Figure C.1 — Graphical representation of elements used for calculation of the variogram and the correlogram

The values of the variance $V_{\text{exp}}(t)$ and the coefficient of correction $r_{\text{exp}}(t)$ at lag t can be calculated from the Equations (5) and (6) in 7.4.

C.3 Blast furnace data

In Tables C.1 and C.2, serial data of some of the important indicators of blast furnace operation are shown. Data are collected for two terms, in May [Series a in Table C.1] and in October [Series b in Table C.2], 1987 from a blast furnace. Production rate is a mass of molten iron (tonnes) produced from a tap hole at one tapping. The tap hole of the blast furnace is opened on the stroke of the hour and separated by 3 h intervals, e.g. 7:00, 10:00, 13:00, 16:00, etc. These times right on the hour are the target and sometimes there is a delay of a few minutes in opening the tap hole due to the difficulty of drilling it. The tap hole is closed just before the molten iron starts to be splashed by the inner air pressure of the furnace. The production rate (P_{mi}), silicon (w_{Si}) and sulfur (w_{Su}) contents representative to each tap are listed in Tables C.1 and C.2.

Table C.1 — Blast furnace data — Series *a*

No.	$P_{\text{mi}}(t)$	w_{Si}(%)	w_{Su}(%)	No.	$P_{\text{mi}}(t)$	w_{Si}(%)	w_{Su}(%)
1	1 183,7	0,13	0,037	46	1 091,3	0,22	0,028
2	1 139,8	0,10	0,052	47	1 191,6	0,23	0,026
3	952,4	0,19	0,033	48	679,0	0,34	0,018
4	1 234,4	0,22	0,055	49	1 611,1	0,32	0,016
5	491,8	0,45	0,013	50	937,0	0,18	0,030
6	949,1	0,25	0,030	51	1 233,8	0,20	0,025
7	881,0	0,13	0,049	42	834,5	0,16	0,036
8	1 308,5	0,14	0,053	53	1 164,7	0,22	0,021
9	795,3	0,25	0,020	54	856,5	0,31	0,021
10	1 133,4	0,23	0,028	55	985,3	0,18	0,025
11	467,6	0,15	0,049	56	840,0	0,16	0,029
12	1 158,8	0,22	0,026	57	938,7	0,14	0,028
13	1 246,2	0,21	0,032	58	1 010,0	0,16	0,026
14	1 177,7	0,24	0,025	59	1 308,1	0,19	0,023
15	943,5	0,19	0,035	60	652,2	0,34	0,019
16	1 233,7	0,22	0,022	61	1 109,8	0,18	0,027
17	648,9	0,15	0,045	62	939,3	0,28	0,023
18	1 483,9	0,17	0,022	63	1 137,2	0,23	0,026
19	810,5	0,22	0,033	64	776,6	0,24	0,018
20	1 011,2	0,25	0,017	65	654,1	0,18	0,021
21	757,9	0,19	0,046	66	1 379,0	0,22	0,035
22	1 101,5	0,29	0,016	67	1 275,5	0,31	0,019
23	672,9	0,28	0,020	68	1 376,2	0,25	0,028
24	1 197,5	0,21	0,026	69	1 023,3	0,31	0,018
25	1 169,3	0,29	0,018	70	827,0	0,45	0,019
26	756,5	0,15	0,040	71	1 322,0	0,26	0,025
27	806,9	0,32	0,019	72	913,3	0,34	0,015
28	906,0	0,20	0,032	73	1 405,4	0,32	0,020
29	1 179,6	0,30	0,018	74	1 522,0	0,30	0,022
30	1 048,1	0,19	0,042	75	926,7	0,31	0,017
31	1 133,4	0,22	0,022	76	952,7	0,31	0,018
32	1 361,2	0,18	0,038	77	884,4	0,27	0,022
33	879,4	0,25	0,023	78	996,3	0,12	0,044
34	961,5	0,19	0,032	79	1 061,8	0,24	0,023
35	1 196,0	0,22	0,023	80	826,0	0,40	0,018
36	999,5	0,17	0,027	81	893,3	0,40	0,018
37	1 065,5	0,15	0,029	82	1 130,6	0,20	0,027
38	1 187,2	0,18	0,030	83	1 058,0	0,20	0,034
39	957,1	0,15	0,028	84	1 099,8	0,42	0,018
40	713,2	0,11	0,037	85	889,0	0,31	0,025
41	1 107,5	0,20	0,023	86	946,1	0,36	0,021
42	880,7	0,13	0,045	87	1 118,0	0,27	0,026
43	1 534,2	0,15	0,033	88	928,1	0,36	0,018
44	942,9	0,12	0,035	89	1 136,7	0,27	0,023
45	992,7	0,21	0,031	90	1 605,5	0,21	0,030

Table C.2 — Blast furnace data — Series b

No.	$P_{\text{mi}}(t)$	$w_{\text{Si}}(\%)$	$w_{\text{Su}}(\%)$	No.	$P_{\text{mi}}(t)$	$w_{\text{Si}}(\%)$	$w_{\text{Su}}(\%)$
1	1 088,1	0,32	0,023	46	1 041,4	0,30	0,014
2	769,3	0,32	0,014	47	1 188,3	0,18	0,027
3	1 665,8	0,30	0,025	48	1 159,7	0,17	0,022
4	1 191,8	0,34	0,016	49	1 423,8	0,24	0,026
5	1 194,9	0,20	0,020	50	1 262,6	0,27	0,017
6	1 040,6	0,27	0,024	51	937,5	0,29	0,016
7	1 542,2	0,16	0,027	42	846,2	0,14	0,026
8	1 012,1	0,20	0,027	53	1 066,8	0,21	0,020
9	1 770,4	0,19	0,027	54	991,2	0,21	0,024
10	913,4	0,32	0,014	55	985,6	0,15	0,025
11	976,6	0,16	0,036	56	877,0	0,13	0,028
12	1 033,9	0,30	0,017	57	1 201,4	0,27	0,017
13	1 299,4	0,25	0,027	58	974,9	0,23	0,023
14	1 156,7	0,37	0,012	59	1 071,0	0,28	0,020
15	1 260,5	0,22	0,026	60	726,2	0,21	0,027
16	940,7	0,35	0,013	61	642,6	0,20	0,021
17	1 488,2	0,32	0,017	62	1 098,2	0,18	0,022
18	1 630,9	0,22	0,019	63	1 188,4	0,15	0,028
19	1 127,1	0,29	0,020	64	1 016,9	0,20	0,023
20	1 123,2	0,14	0,035	65	911,7	0,22	0,025
21	1 087,2	0,26	0,022	66	1 038,1	0,32	0,015
22	963,2	0,18	0,022	67	746,2	0,21	0,026
23	1 446,9	0,25	0,019	68	1 231,7	0,18	0,025
24	1 038,7	0,16	0,027	69	1 261,2	0,21	0,021
25	956,0	0,23	0,025	70	1 102,5	0,28	0,022
26	846,7	0,21	0,022	71	1 222,3	0,19	0,030
27	732,4	0,27	0,030	72	1 118,5	0,29	0,023
28	1 007,1	0,18	0,026	73	1 051,8	0,27	0,030
29	1 039,6	0,23	0,019	74	824,0	0,24	0,020
30	1 197,0	0,19	0,021	75	1 053,2	0,16	0,029
31	1 093,1	0,20	0,025	76	1 218,9	0,19	0,022
32	1 178,4	0,16	0,027	77	1 449,1	0,22	0,026
33	1 098,7	0,16	0,026	78	888,2	0,18	0,026
34	769,4	0,12	0,028	79	1 172,1	0,12	0,035
35	1 071,7	0,19	0,031	80	1 273,2	0,32	0,017
36	787,6	0,22	0,023	81	1 554,6	0,18	0,029
37	1 035,2	0,24	0,020	82	1 287,5	0,25	0,019
38	1 017,2	0,38	0,025	83	1 526,3	0,26	0,019
39	1 087,0	0,35	0,014	84	1 224,6	0,30	0,013
40	848,3	0,28	0,018	85	1 370,8	0,22	0,023
41	1 324,5	0,38	0,015	86	1 128,1	0,16	0,021
42	991,1	0,35	0,014	87	1 043,3	0,21	0,024
43	1 017,5	0,28	0,018	88	952,4	0,34	0,020
44	1 086,9	0,27	0,015	89	1 550,1	0,26	0,022
45	1 329,2	0,17	0,031	90	1 192,6	0,40	0,014

Table C.3 gives the variogram values for series $P_{\mathrm{mi}}(a)$ and $P_{\mathrm{mi}}(b)$, $w_{\mathrm{Si}}(a)$ and $w_{\mathrm{Si}}(b)$ and $w_{\mathrm{Su}}(a)$ and $w_{\mathrm{Su}}(b)$ at lag t (t running from 0 to 18) are shown.

Table C.3 — Variogram values of blast furnace data

Lag t	$P_{\mathrm{mi}}(a)$ $\times 10^{-4}$	$P_{\mathrm{mi}}(b)$ $\times 10^{-4}$	$w_{\mathrm{Si}}(a)$ $\times 10^{2}$	$w_{\mathrm{Si}}(b)$ $\times 10^{2}$	$w_{\mathrm{Su}}(a)$ $\times 10^{4}$	$w_{\mathrm{Su}}(b)$ $\times 10^{4}$
0	0	0	0	0	0	0
1	6,618	4,448	0,398	0,336	0,922	0,326
2	4,642	3,978	0,490	0,278	0,639	0,183
3	6,590	4,922	0,537	0,410	0,875	0,327
4	4,907	3,621	0,447	0,381	0,510	0,235
5	5,870	4,601	0,436	0,500	0,710	0,340
6	3,816	3,430	0,466	0,477	0,717	0,315
7	6,494	5,589	0,451	0,466	0,695	0,286
8	4,571	4,009	0,490	0,422	0,770	0,280
9	7,150	4,037	0,520	0,512	0,611	0,332
10	5,070	4,249	0,482	0,470	0,702	0,310
11	6,275	4,915	0,469	0,435	0,648	0,299
12	4,469	4,195	0,512	0,456	0,810	0,276
13	6,696	4,526	0,464	0,406	0,533	0,285
14	5,126	3,518	0,446	0,501	0,800	0,265
15	5,897	4,664	0,497	0,404	0,646	0,287
16	4,565	5,354	0,495	0,466	0,836	0,281
17	4,880	5,170	0,517	0,476	0,677	0,308
18	5,180	4,577	0,523	0,511	0,879	0,282

In Table C.4, correlogram values of $P_{\mathrm{mi}}(a)$ and $P_{\mathrm{mi}}(b)$, $w_{\mathrm{Si}}(a)$ and $w_{\mathrm{Si}}(b)$ and $w_{\mathrm{Su}}(a)$ and $w_{\mathrm{Su}}(b)$ ($\times 10^4$) at lag t (t running from 0 to 18) are shown, indicating significance levels at 5 % and 1 %.

Table C.4 — Correlogram values of blast furnace data

Lag t	$P_{\mathrm{mi}}(a)$	$P_{\mathrm{mi}}(b)$	$w_{\mathrm{Si}}(a)$	$w_{\mathrm{Si}}(b)$	$w_{\mathrm{Su}}(a)$	$w_{\mathrm{Su}}(b)$
0	1,000	1,000	1,000	1,000	1,000	1,000
1	−0,256[a]	0,103	0,344[b]	0,214[a]	−0,030	−0,142
2	0,127	0,179	0,188	0,351[b]	0,266[a]	0,358[b]
3	−0,228[a]	−0,046	0,106	0,033	−0,002	−0,137
4	0,091	0,238[a]	0,264[a]	0,098	0,397[b]	0,185
5	−0,110	0,043	0,244[a]	−0,183	0,152	−0,167
6	0,285[b]	0,288[b]	0,196	−0,117	0,153	−0,071
7	−0,206	−0,173	0,197	−0,093	0,159	0,019
8	0,154	0,149	0,129	0,020	0,029	0,043
9	−0,317[b]	0,097	0,083	−0,178	0,242[a]	−0,127
10	0,075	0,029	0,137	−0,083	0,132	−0,067
11	−0,181	−0,116	0,142	−0,010	0,182	−0,061
12	0,169	0,059	0,074	−0,073	−0,025	−0,010
13	−0,237[a]	−0,015	0,159	0,053	0,334[b]	−0,039
14	0,060	0,207	0,203	−0,186	−0,015	0,017
15	−0,069	−0,045	0,116	0,053	0,196	−0,052
16	0,179	−0,188	0,126	−0,109	−0,054	−0,052
17	0,092	−0,173	0,091	−0,130	0,152	−0,145
18	0,006	−0,073	0,084	−0,202	−0,118	−0,053

[a] Significance level 5 %

[b] Significance level 1 %

Figures C.2, C.4 and C.6 show variograms for $P_{\mathrm{mi}}(a)$ and $P_{\mathrm{mi}}(b)$, $w_{\mathrm{Si}}(a)$ and $w_{\mathrm{Si}}(b)$, and $w_{\mathrm{Su}}(a)$ and $w_{\mathrm{Su}}(b)$, respectively. Figures C.3, C.5 and C.7 show correlograms of $P_{\mathrm{mi}}(a)$ and $P_{\mathrm{mi}}(b)$, $w_{\mathrm{Si}}(a)$ and $w_{\mathrm{Si}}(b)$ and $w_{\mathrm{Su}}(a)$ and $w_{\mathrm{Su}}(b)$, respectively.

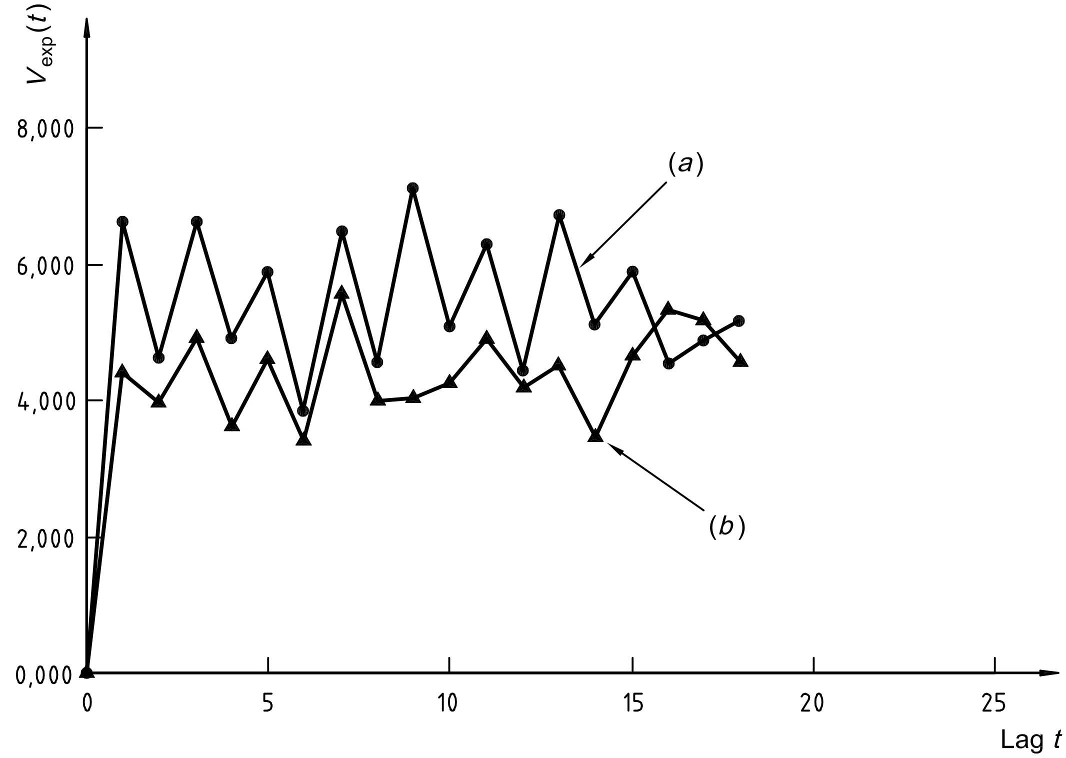


Figure C.2 — Variogram of $P_{\mathrm{mi}}(a)$ and $P_{\mathrm{mi}}(b)$

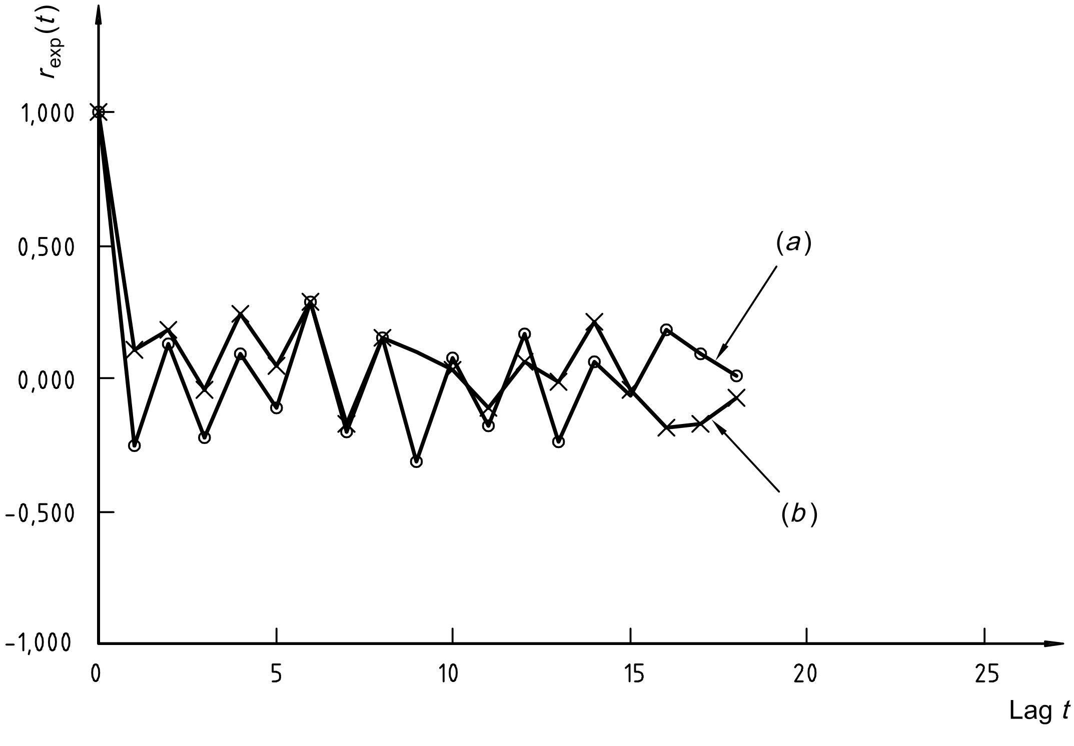


Figure C.3 — Correlogram of $P_{\mathrm{mi}}(a)$ and $P_{\mathrm{mi}}(b)$

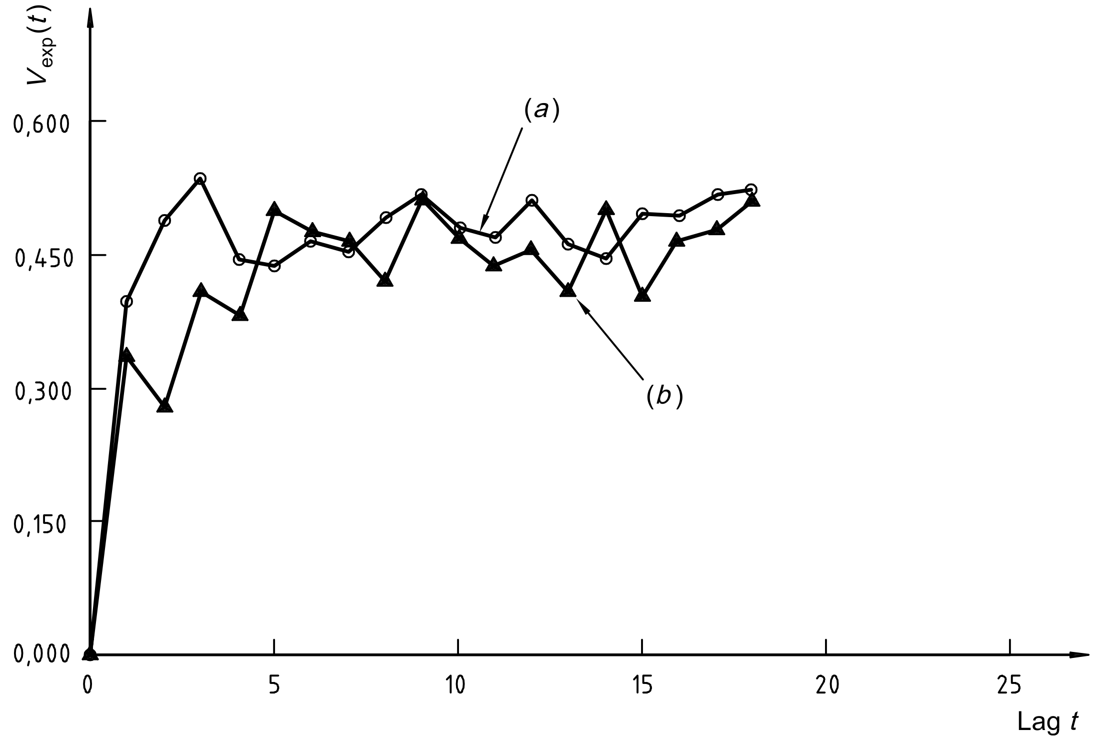


Figure C.4 — Variogram of $w_{Si}(a)$ and $w_{Si}(b)$

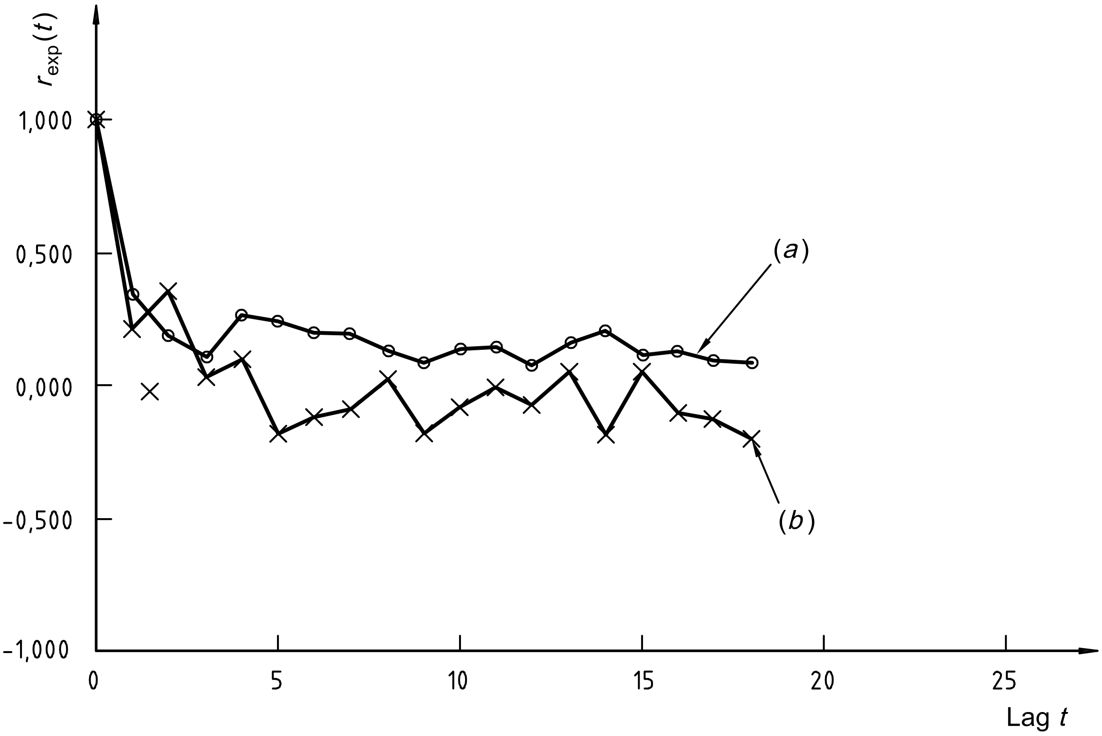


Figure C.5 — Correlogram of $w_{Si}(a)$ and $w_{Si}(b)$

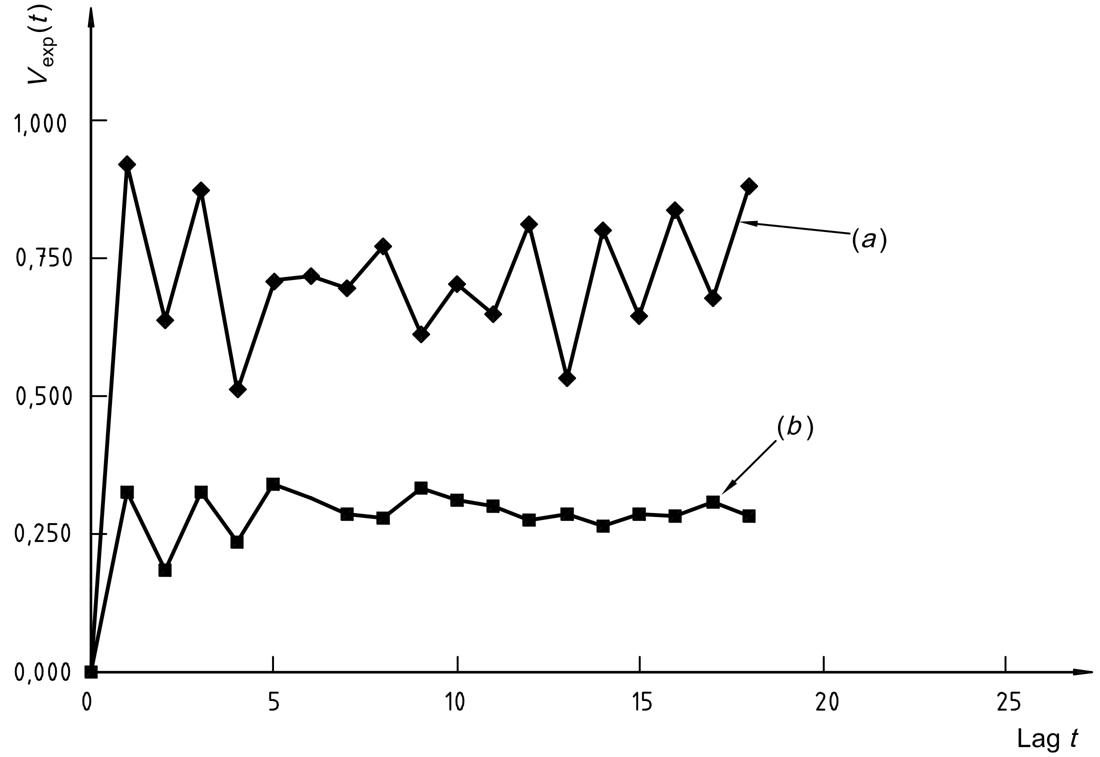


Figure C.6 — Variogram of $w_{\mathrm{Su}}(a)$ and $w_{\mathrm{Su}}(b)$

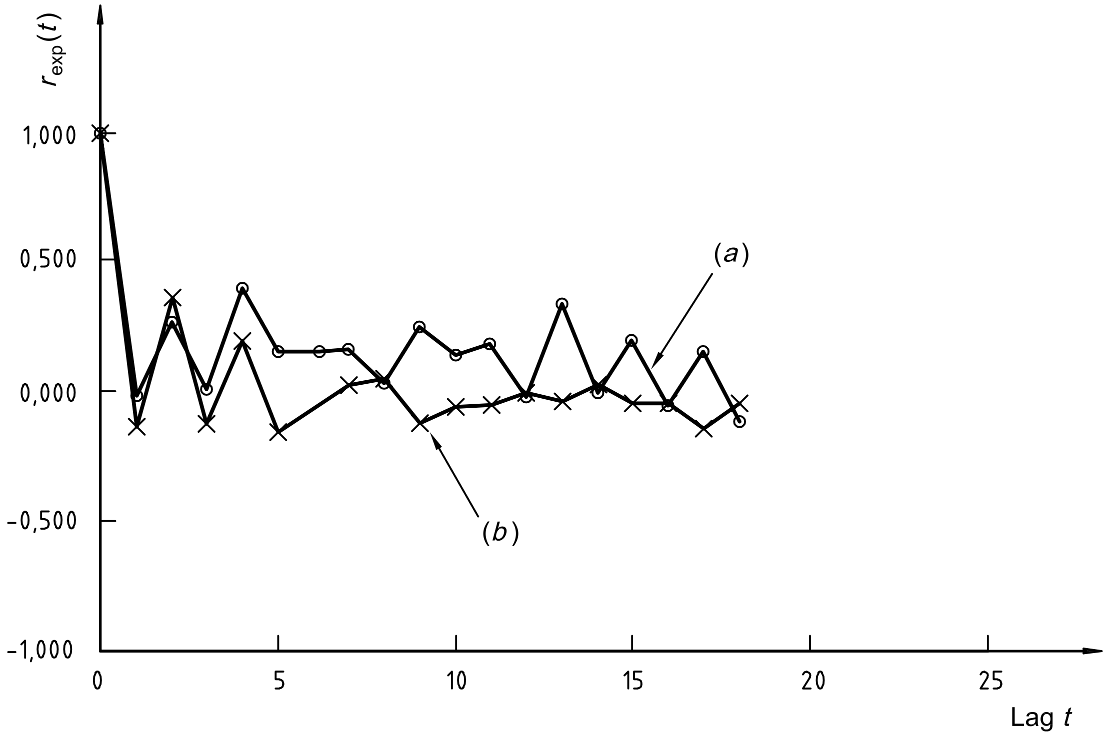


Figure C.7 — Correlogram of $w_{\mathrm{Su}}(a)$ and $w_{\mathrm{Su}}(b)$

Figure C.2 shows a special movement which looks like a saw edge. Figure C.6 shows also a similar movement, strongly for small lags. The variogram in Figure C.2 may be subdivided as follows:

$$V_{\mathrm{exp}}(t) = V_{\mathrm{r}} = V_{\mathrm{c}} \tag{C.3}$$

or

$$V_{\mathrm{exp}}(t) = (-1)^{t+1} V_{\mathrm{a}} \tag{C.4}$$

where

V_{r} is the variance of random variation;

V_{c} is the variance of cyclic variation;

V_{a} is the variance corresponding to the amplitude of cyclic variation.

In this special case, $V_{\mathrm{exp}}(t)$ for series P_{mi} may be approximated using the values given in Figure C.2 with the following equation:

$$V_{\mathrm{exp}}(t) = 5{,}2 + (-1)^{t+1} \times 1{,}0 \tag{C.5}$$

However, in general, it is not so easy to subdivide this variance into individual patterns of variation.

It is partially assumed that the cyclic movement shown in Figure C.2 is caused by the starting time of opening of tap hole (on the hour), irrespective of the previous closing time of tap hole. However, further dimensions of this movement into physical interpretations and actions to be taken should be left to the engineers who are working for the blast furnace operation and for the subsequent de-silicon (including de-sulfur) plant. This example is of interest in that the statistical analysis of serial data provides useful information which cannot be obtained using the conventional methods of statistical analysis.

Figure C.8 shows the relationship between the variogram and the correlogram of $P_{\mathrm{mi}}(a)$. In this figure, the correlogram is reflected on the axis $r = +1$. That is, it is shown as $[1 - r_{\mathrm{exp}}(t)]$, and similar movements of the graph of $[1 - r_{\mathrm{exp}}(t)]$ to the variogram $V_{\mathrm{exp}}(t)$ are displayed. Between the variogram and the correlogram, the following relationship exists:

$$V_{\mathrm{exp}}(t) = \sigma_{\mathrm{t}}^{2} \left[-r_{\mathrm{exp}}(t) \right] \tag{C.6}$$

where σ_{t}^{2} is the total variance of the original series and constant.

As for the series $w_{\mathrm{Su}}(a)$, the variogram $V_{\mathrm{exp}}(t)$ and the graph of $[1 - r_{\mathrm{exp}}(t)]$ demonstrate similar movements as shown in Figure C.9.

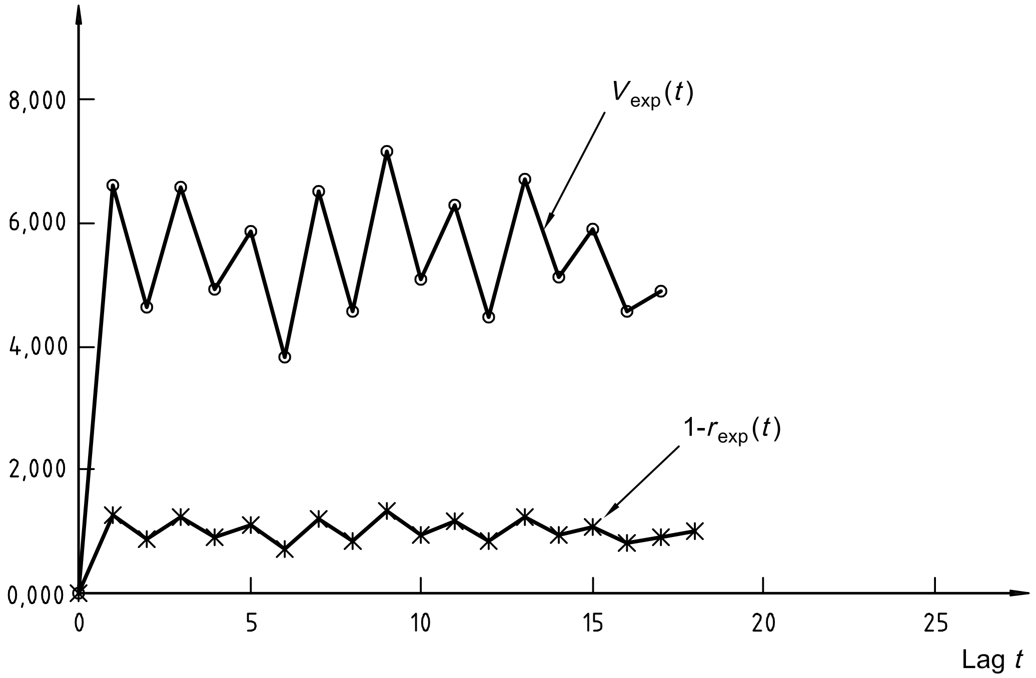


Figure C.8 — Relationship between $V_{\text{exp}}(t)$ **and** $[1 - r_{\text{exp}}(t)]$ **for** $P_{\text{mi}}(a)$

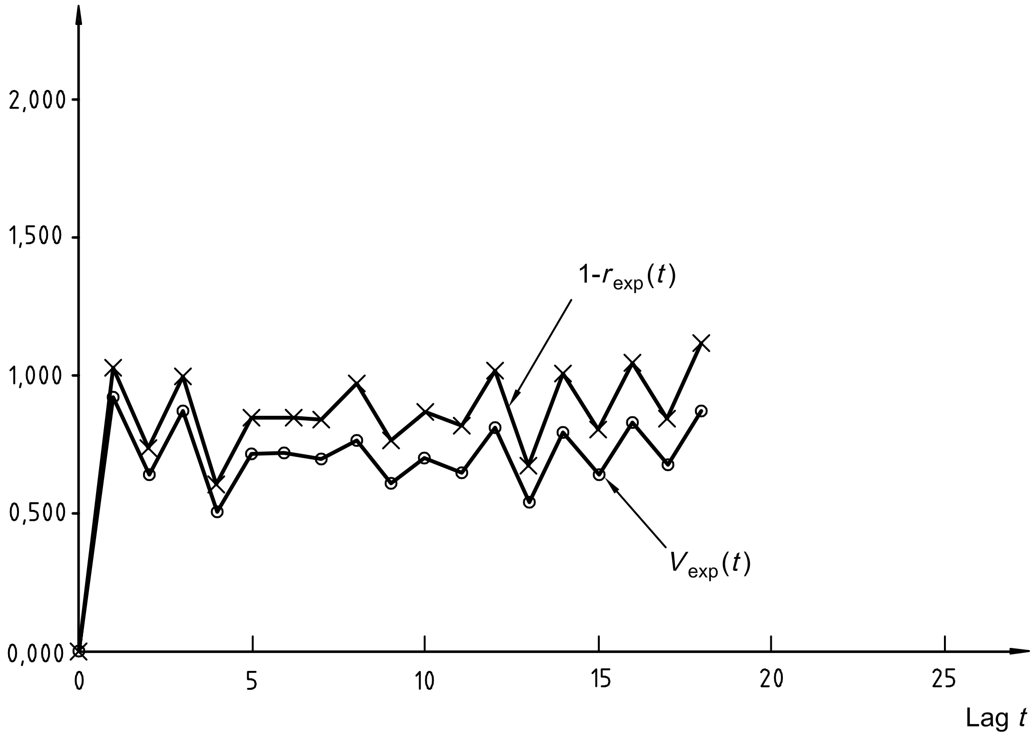


Figure C.9 — Relationship between $V_{\text{exp}}(t)$ **and** $[1 - r_{\text{exp}}(t)]$ **for** $w_{\text{Su}}(a)$

C.4 Thickness of paper

This application provides an example of correlated variations. Thickness of paper (µm) is measured at a constant interval (5 m) along the length of rolled-formed paper and 208 readings are provided as shown is Table C.5.

Table C.5 — Thickness of paper

Values in micrometres

Reading No.	1	2	3	4	5	6	7	8	9	10
0	572	575	568	558	555	564	576	570	570	575
10	587	562	580	573	576	573	585	576	580	576
20	586	571	575	582	570	568	588	560	580	590
30	596	564	605	576	579	579	581	573	570	576
40	573	576	577	575	573	572	562	580	565	572
50	558	569	559	558	556	572	552	560	565	561
60	553	562	555	560	552	561	559	562	550	553
70	556	548	556	536	560	540	548	539	552	543
80	546	541	542	551	538	547	542	541	546	548
90	543	545	547	559	541	543	553	546	550	559
100	548	557	555	559	565	552	555	552	560	562
110	563	563	562	559	581	574	560	583	563	573
120	577	582	573	571	589	588	587	592	582	589
130	589	584	584	599	580	589	575	573	581	576
140	568	570	586	560	570	574	570	556	569	578
150	569	562	576	570	560	572	570	554	568	558
160	562	562	560	547	550	569	547	561	548	556
170	558	549	553	559	564	536	555	558	539	542
180	564	546	557	550	558	550	549	551	541	552
190	542	553	551	558	574	562	561	560	565	569
200	575	568	569	572	565	562	582	570	—	—

Variogram values and correlogram values up to lag $t = 25$ are shown in Table C.6 and the variogram and the correlogram are shown in Figures C.10 and C.11, respectively.

The correlogram shows highly significant correlation up to lag $t = 21$ and significant correlation at lag $t = 22$. Since more than 20 successive data are correlated with each other, the serial data are subdivided into nine groups composed of 20 successive data starting from No. 11 for convenience purposes. Single factor ANOVA shows significant difference between groups. Confidence intervals of 95 % average values of each group are shown in Figure C.12. Rough control of the mixer gate is observed and more frequent control of mixer gate is suggested.

Table C.6 — Variogram and correlogram values of thickness of paper

Lag t	Variogram values	Correlogram values
0	0	1 000[a]
1	62,435	0,674[a]
2	49,638	0,741[a]
3	48,324	0,749[a]
4	48,931	0,747[a]
5	70,569	0,636[a]
6	58,223	0,701[a]
7	64,995	0,667[a]
8	77,583	0,604[a]
9	78,638	0,600[a]
10	81,748	0,586[a]
11	90,165	0,542[a]
12	94,781	0,521[a]
13	99,928	0,496[a]
14	113,487	0,429[a]
15	117,096	0,413[a]
16	122,034	0,389[a]
17	132,296	0,337[a]
18	137,608	0,309[a]
19	142,077	0,286[a]
20	152,202	0,233[a]
21	154,944	0,217[a]
22	165,304	0,166[b]
23	170,103	0,144
24	183,158	0,079
25	184,011	0,077

[a] Highly significant correlation.

[b] Significant correlation.

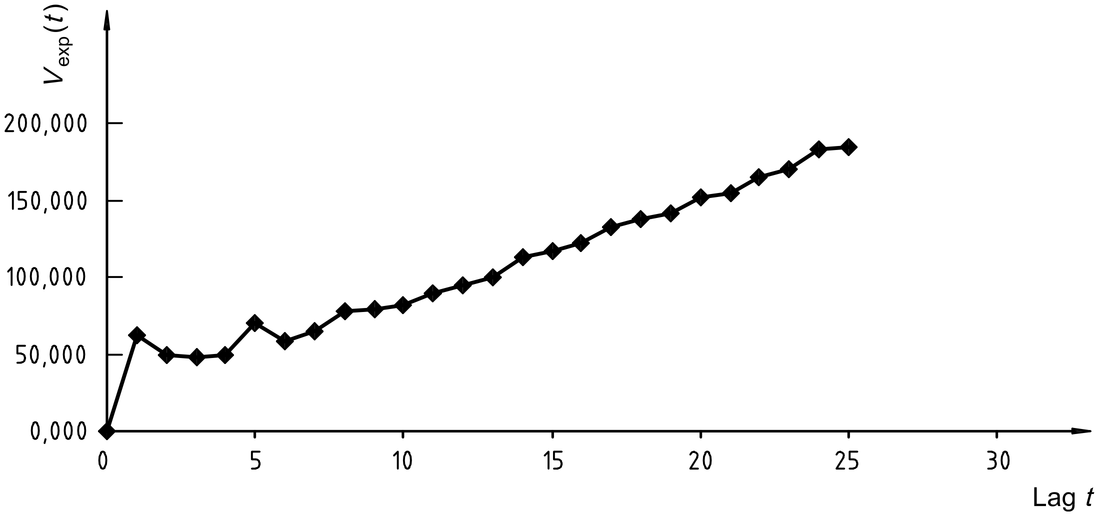


Figure C.10 — Variogram of thickness of paper

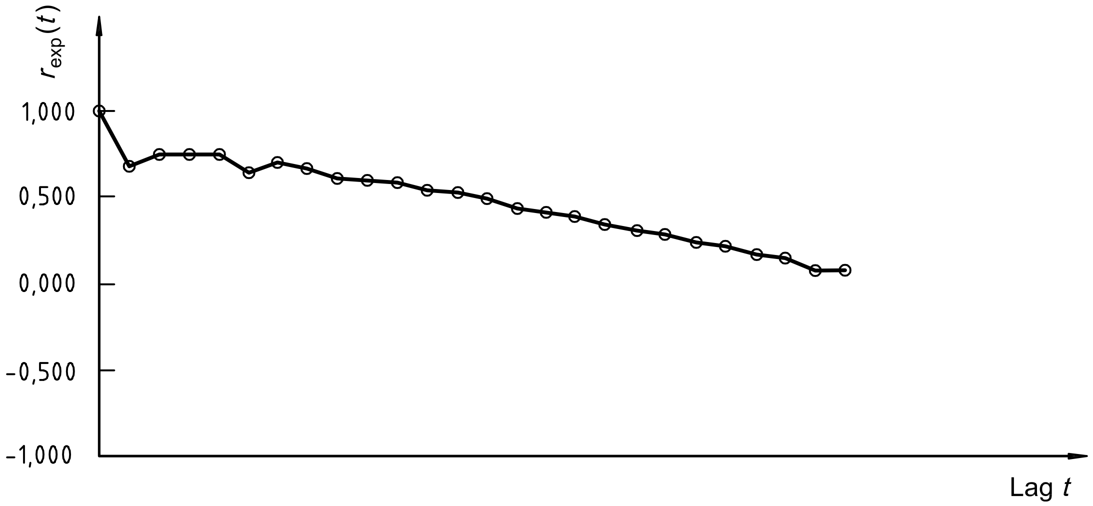


Figure C.11 — Correlogram of thickness of paper

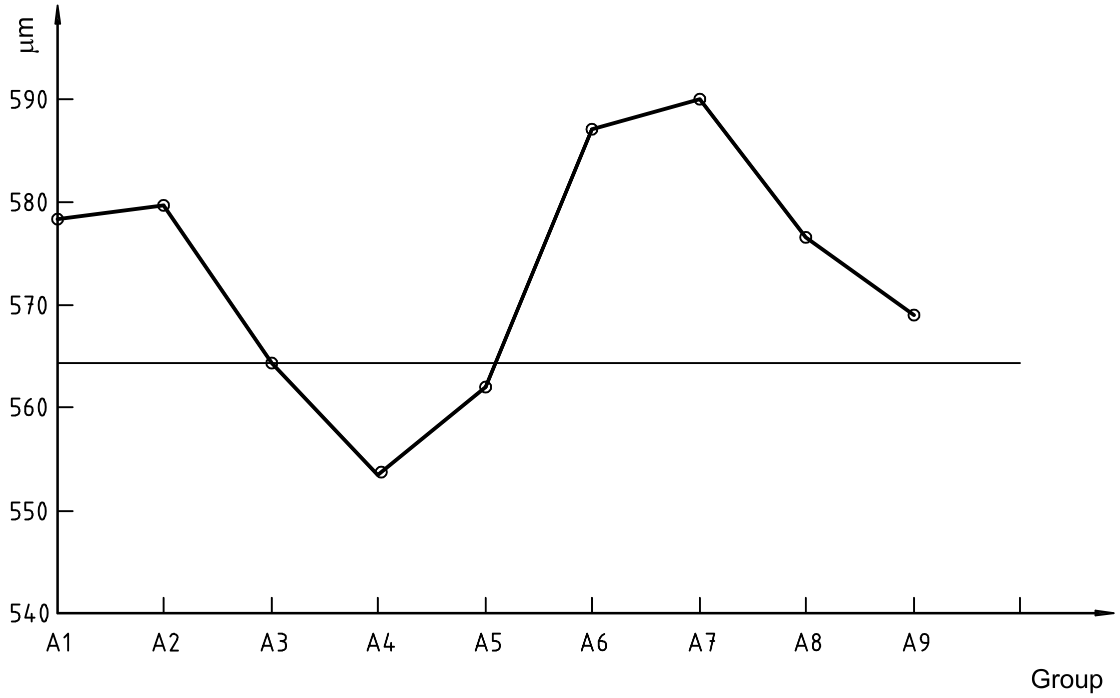


Figure C.12 — Confidence intervals for each group

C.5 Moisture in iron ores

The number of increments taken for the moisture determination of a lot is usually on the average around 50 per lot. Until several years ago, the procedure was to combine several increments (e.g. 5 increments) into samples and measure the moisture on the samples. This has been applied as the usual procedure in most of the steel mills.

Recently, moisture content in iron ores has been determined increment by increment in several steel mills in order to avoid the loss of moisture during storage. Data obtained in such a mill will be dealt with here. Data for iron ores imported to steel mills A and B from the same mine, are grouped either by lumps or by fines. Values of moisture on individual increments from 12 vessels, consist of Lumps A (S1 to S3), Lumps B (S4 to S6), Fines A (S7 to S9) and Fines B (S10 to S12), are shown in Table C.7 and C.8 and are analysed hereafter.

As a typical example, the result of the statistical analysis of Series No. 1 is shown below. A graph of Series No.1 is shown in Figure C.13. In the graph, a trend decreasing with the increase of unloaded quantity is observed. Regression analysis of the values of moisture on the increment number shows a highly significant dependence of moisture on the increment number and the following regression is obtained:

$$w_{\mathrm{m}} = 2{,}164 - 0{,}008\,5i \qquad \text{(C.7)}$$

where

i is the increment number;

w_{m} is the mass fraction, expressed as a percentage, of moisture content on individual increments.

As the number of increments taken from a lot is varied from vessel to vessel, converting the increment number into the unloaded ratio, the following regression equation is obtained:

$$w_{\mathrm{m}} = 2{,}164 - 0{,}51u \qquad \text{(C.8)}$$

where

u is the unloaded ratio.

When the total number of increments taken from a lot is n, the unloaded ratio at the i-th increment is i/n.

Equation (C.8) is expressed in the following general form:

$$w_\mathrm{m} = b_0 + b_1 u \tag{C.9}$$

and values of b_1 for each series are shown in Table C.9.

The variogram of S1 is shown in Figure C.14. In this figure, the “original series” shows the variogram of the original series, while “without outlier No. 19” shows the variogram of the series without outlier No. 19. Note that the outlier pushes up the variogram of the series without the outlier.

Table C.7 — Moisture content of ores (%) for increments S1 to S6

No.	S1	S2	S3	S4	S5	S6
1	2,230	2,573	3,123	2,743	2,744	2,895
2	2,109	2,532	3,040	2,719	2,763	2,966
3	2,263	2,394	2,870	2,912	2,881	2,964
4	1,993	2,679	2,914	2,672	2,988	2,769
5	2,091	2,567	3,118	2,656	2,756	2,833
6	2,012	2,174	2,871	2,582	2,813	2,945
7	2,213	2,421	2,934	2,780	2,795	2,852
8	2,049	2,350	2,515	3,090	2,948	2,826
9	2,080	2,381	2,705	2,861	2,943	2,728
10	2,111	2,557	2,755	2,801	3,022	2,665
11	2,004	2,465	2,975	2,559	3,012	2,569
12	1,993	2,373	2,921	2,567	2,74	2,808
13	2,110	2,417	2,918	2,680	2,723	2,775
14	2,211	2,449	2,730	2,788	2,329	3,020
15	2,116	2,489	3,631	2,694	2,803	2,850
16	2,018	2,528	2,798	2,481	2,602	2,679
17	1,970	2,394	2,841	2,434	2,635	2,776
18	1,732	2,297	2,812	2,435	2,603	2,824
19	2,515	2,306	2,639	2,445	2,551	2,644
20	1,941	2,447	2,495	2,732	2,572	2,555
21	1,769	2,379	2,571	2,674	2,507	2,600
22	1,963	2,316	2,646	2,738	2,575	2,622
23	1,607	2,242	2,447	2,739	2,531	2,442
24	1,829	2,310	2,484	2,573	2,488	2,353
25	1,891	2,378	2,528	2,435	2,612	2,274
26	1,691	2,331	2,874	2,760	2,602	2,273
27	1,969	2,342	2,524	2,549	2,625	2,396
28	2,240	2,368	2,504	2,547	2,756	2,282
29	2,000	2,394	2,603	2,522	2,723	2,333
30	2,000	2,438	2,395	2,815	2,572	2,715
31	1,857	2,395	2,109	2,363	2,504	2,564
32	1,874	2,330	2,261	2,347	2,315	2,516
33	1,836	2,392	2,412	2,393	2,460	2,597
34	1,776	2,569	2,426	2,804	2,425	5,729
35	1,858	2,174	2,398	2,584	2,354	2,554
36	1,906	2,386	2,365	2,501	2,463	2,497
37	1,919	2,180	2,669	2,367	2,590	2,723
38	2,018	2,276	2,697	2,217	2,212	2,762
39	1,749	2,179	2,711	2,668	2,304	2,278
40	1,982	2,398	2,591	2,485	2,271	2,432
41	1,986	2,339	2,734	2,306	2,454	2,527
42	1,693	2,507	2,351	2,326	2,368	2,580
43	1,643	2,281	2,551	2,076	2,117	—
44	1,889	2,144	2,297	2,136	2,165	—
45	1,898	1,999	2,269	2,701	2,157	—
46	2,003	1,999	2,510	—	1,979	—
47	1,891	2,190	2,409	—	—	—
48	1,849	2,201	—	—	—	—
49	1,928	2,265	—	—	—	—
50	1,961	2,203	—	—	—	—
51	1,760	1,984	—	—	—	—
52	1,734	2,239	—	—	—	—
53	1,511	2,429	—	—	—	—
54	1,822	2,280	—	—	—	—
55	1,701	1,977	—	—	—	—
56	1,592	2,141	—	—	—	—
57	1,482	2,012	—	—	—	—
58	1,505	—	—	—	—	—
59	1,493	—	—	—	—	—
60	1,540	—	—	—	—	—

Table C.8 — Moisture content of ores (%) for increments S7 to S12

No.	S7	S8	S9	S10	S11	S12
1	4,625	5,554	4,535	4,595	4,710	4,992
2	4,617	5,303	4,505	4,579	4,660	5,066
3	4,558	5,267	4,605	4,559	4,696	4,911
4	4,643	5,261	4,462	4,544	4,681	4,998
5	4,623	5,144	4,530	4,585	4,656	5,024
6	4,656	5,197	4,482	4,574	4,684	5,061
7	4,586	5,193	4,603	4,572	4,679	4,995
8	4,638	5,171	4,626	4,633	4,689	4,976
9	4,474	5,159	4,503	4,616	4,633	4,982
10	4,687	5,161	4,426	4,693	4,685	4,977
11	4,600	5,000	4,432	4,483	4,758	4,976
12	4,659	5,292	4,437	4,559	4,691	5,042
13	4,659	5,000	4,507	4,528	4,709	1,912
14	4,553	5,127	4,542	4,543	4,690	4,893
15	4,591	5,108	4,569	4,571	4,649	4,982
16	4,630	4,828	4,564	4,500	4,668	4,883
19	4,590	4,951	4,535	4,530	4,722	4,936
20	4,617	5,019	4,505	4,488	4,594	4,956
21	4,603	5,148	4,564	4,404	4,605	4,880
22	4,576	5,123	4,573	4,610	4,669	4,899
23	4,585	5,079	4,564	4,474	4,615	4,992
24	4,603	4,991	4,456	4,456	4,659	5,115
25	4,635	5,095	4,480	4,418	4,601	4,959
26	5,285	4,972	4,503	4,427	4,572	4,997
27	5,026	5,017	4,385	4,486	4,537	4,971
28	4,929	5,005	4,454	4,467	4,588	4,950
29	5,000	5,050	4,478	4,464	4,564	4,960
30	4,000	4,979	4,411	4,450	4,566	4,997
31	4,868	5,029	4,434	4,634	4,577	4,988
32	4,733	4,977	4,521	4,535	4,637	4,963
33	4,597	5,011	4,478	4,528	4,458	4,996
34	4,733	5,030	—	4,450	4,714	4,945
35	4,496	5,019	—	4,458	4,648	5,041
36	4,584	5,025	—	4,424	4,644	4,981
37	4,671	5,113	—	4,571	4,698	5,029
38	4,723	4,849	—	4,456	4,689	4,885
39	4,688	5,069	—	4,501	4,638	4,975
40	4,570	—	—	4,544	4,627	4,866
41	4,522	—	—	4,390	4,659	4,659
42	4,332	—	—	4,466	4,599	4,788
43	4,584	—	—	4,467	4,637	4,891
44	4,557	—	—	4,391	4,677	4,778
45	4,607	—	—	4,405	4,647	4,969
46	4,537	—	—	—	4,649	4,843
47	4,534	—	—	—	4,515	4,907
48	4,459	—	—	—	4,579	4,942
49	4,571	—	—	—	4,553	4,945
50	4,684	—	—	—	4,561	4,915
51	4,548	—	—	—	—	4,913
52	4,568	—	—	—	—	4,937
53	4,368	—	—	—	—	4,947
54	4,524	—	—	—	—	4,839
55	4,320	—	—	—	—	4,950
56	4,479	—	—	—	—	4,818
57	4,643	—	—	—	—	

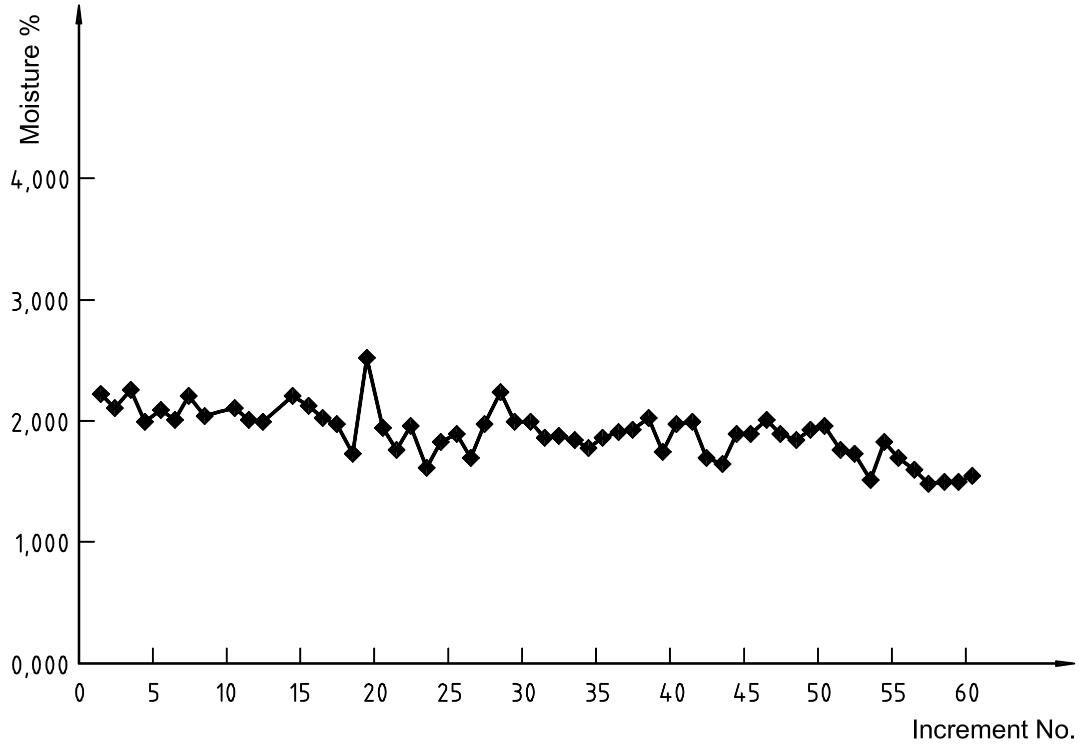
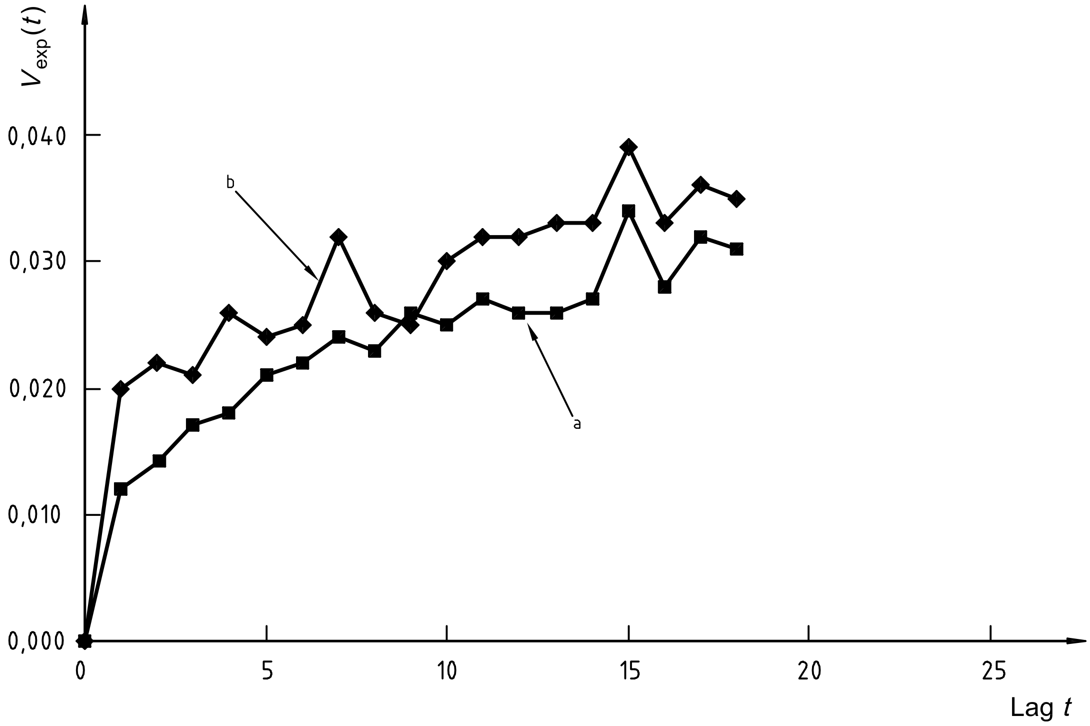


Figure C.13 — Graph of moisture content for Series No.1



[a] Without outlier No. 19

[b] Original series

Figure C.14 — Variogram for Series No.1

Variogram values for series S1 to S6 and S7 to S12 are listed in Tables C.10 and C.11, and variograms are shown in Figures C.15 to C.16 respectively. Since S1 to S6 and S7 to S12 are the same varieties of iron ores respectively, it is important that variograms in these figures agree to some extent.

Results of statistical analysis on each series are summarized in the Table C.9. Table C.9 shows that significant trends are observed in most of the series, and the regression slopes are all negative. The slopes for lumps are larger than those for fines, whereas the levels of moisture content of lumps are lower than those of fines. Consider a part of a lot corresponding to two adjacent increments as a stratum. The quality variation within strata can be estimated from the variogram values for $t = 1$, as shown in Table C.9.

Additional data are provided by steel mill C, which is located in an area under severe dust control regulations for "sake" plant. Imported iron ores are sprayed heavily with water during unloading. The same variety of iron as steel mills A and B will be dealt with below. Values of moisture on increments taken from six vessels, which consist of three vessels of lumps (S13 to S15) and three vessels of fines (S16 to S18), are shown in Table C.12. Regression analysis, if necessary excluding outlier values, of moisture content of the increment number shows almost none of the tendency that was observed in the data of A and B. Variogram values are shown in Table C.13. Variogram values of lag $t = 1$ are more than 10 times greater than those of ore not sprayed with water. Variograms of S13 to S18 are shown in Figure C.17. Results of statistical analysis on the data obtained from the mill C are also summarized in Table C.9.

Table C.9 — Summary of statistical analysis for moisture in iron ores

Kind of ore	Steel mills	Series No.	Number of increments	Regression analysis b_1	Variogram $V_{\text{exp}}(t = 1)$
Lumps	A	1	60	– 0,51[a]	0,012
	A	2	57	– 0,36[a]	0,012
	A	3	47	– 0,37[a]	0,016
	B	4	45	– 0,27[a]	0,022
	B	5	46	– 0,46[a]	0,012
	B	6	42	– 0,47[a]	0,011
	C	13	48	– 0,09[a]	0,134
	C	14	54	0	0,110
	C	15	50	0	0,166
Fines	A	7	57	0	0,023
	A	8	40	0	0,007
	A	9	33	– 0,07[b]	0,002
	B	10	45	– 0,09[a]	0,003
	B	11	50	– 0,10[a]	0,002
	B	12	56	– 0,11[a]	0,004
	C	16	33	0	0,075
	C	17	48	0	0,017
	C	18	47	0	0,206

[a] Highly significant correlation.

[b] Significant correlation.

Table C.10 — Variogram values for increments S1 to S6

Lag *t*	**S1**	**S1R**[a]	**S2**	**S3**	**S3R**[a]	**S4**	**S5**	**S6**	**S6R**[a]
0	0	0	0	0	0	0	0	0	0
1	0,020	0,012	0,012	0,032	0,016	0,022	0,012	0,254	0,011
2	0,022	0,014	0,015	0,033	0,022	0,032	0,015	0,278	0,019
3	0,021	0,017	0,017	0,039	0,026	0,034	0,020	0,262	0,018
4	0,026	0,018	0,016	0,043	0,027	0,031	0,022	0,254	0,020
5	0,024	0,021	0,015	0,056	0,033	0,024	0,023	0,339	0,025
6	0,025	0,022	0,011	0,059	0,038	0,020	0,024	0,342	0,026
7	0,032	0,024	0,015	0,064	0,040	0,031	0,025	0,334	0,031
8	0,026	0,023	0,016	0,057	0,035	0,035	0,028	0,352	0,037
9	0,025	0,026	0,015	0,058	0,034	0,037	0,035	0,219	0,037
10	0,030	0,025	0,014	0,050	0,032	0,036	0,038	0,218	0,040
11	0,032	0,027	0,014	0,041	0,028	0,031	0,036	0,213	0,042
12	0,032	0,026	0,014	0,056	0,031	0,027	0,039	0,200	0,040
13	0,033	0,026	0,017	0,058	0,039	0,030	0,043	0,210	0,041
14	0,033	0,027	0,019	0,060	0,043	0,036	0,044	0,224	0,043
15	0,039	0,034	0,021	0,066	0,049	0,037	0,051	0,217	0,042
16	0,033	0,028	0,018	0,084	0,059	0,039	0,052	0,202	0,044
17	0,036	0,032	0,021	0,083	0,067	0,043	0,054	0,224	0,052
18	0,035	0,031	0,019	0,095	0,072	0,042	0,051	0,248	0,054

[a] R after a series numbers means “without outlier”.

Table C.11 — Variogram values for increments S7 to S12

Lag t	S7	S7R[a]	S8	S8R[a]	S9	S10	S11	S12
0	0	0	0	0	0	0	0	0
1	0,025	0,023	0,008	0,007	0,002	0,003	0,002	0,004
2	0,025	0,021	0,006	0,005	0,003	0,003	0,002	0,004
3	0,028	0,025	0,006	0,005	0,004	0,003	0,002	0,004
4	0,039	0,023	0,010	0,008	0,004	0,004	0,003	0,005
5	0,025	0,019	0,008	0,007	0,004	0,004	0,003	0,005
6	0,028	0,021	0,009	0,008	0,003	0,004	0,003	0,005
7	0,031	0,022	0,011	0,009	0,003	0,004	0,003	0,006
8	0,033	0,027	0,010	0,008	0,003	0,004	0,003	0,006
9	0,036	0,026	0,012	0,010	0,004	0,003	0,004	0,007
10	0,034	0,024	0,014	0,009	0,004	0,004	0,004	0,006
11	0,032	0,024	0,011	0,010	0,004	0,005	0,004	0,007
12	0,031	0,021	0,017	0,012	0,004	0,005	0,004	0,006
13	0,036	0,029	0,014	0,011	0,003	0,005	0,004	0,005
14	0,040	0,029	0,018	0,015	0,003	0,005	0,004	0,007
15	0,044	0,032	0,020	0,009	0,003	0,005	0,004	0,006
16	0,041	0,030	0,017	0,012	0,003	0,006	0,004	0,006
17	0,045	0,032	0,017	0,012	0,003	0,005	0,004	0,007
18	0,041	0,028	0,019	0,012	0,003	0,005	0,003	0,006

[a] R after a series numbers means “without outlier”.

Table C.12 — Moisture content of ores (%) for increments S13 to S18

No.	S13	S14	S15	S16	S17	S18
1	3,299	3,432	3,532	5,293	5,080	6,442
2	3,451	2,585	2,885	5,363	4,897	6,184
3	2,833	3,226	3,270	5,406	5,040	5,157
4	2,708	3,049	3,051	5,368	4,949	5,036
5	3,646	2,880	2,404	5,417	4,912	5,435
6	3,231	2,964	2,592	5,837	5,125	4,995
7	2,205	2,424	2,603	5,083	4,986	5,008
8	2,763	2,403	2,320	5,002	5,074	5,230
9	3,294	2,331	2,413	5,415	5,032	6,290
10	2,423	2,402	2,570	5,467	5,016	4,942
11	2,671	2,602	2,421	5,126	5,054	4,716
12	3,506	2,517	2,792	5,262	5,024	5,014
13	3,417	2,757	2,658	5,038	4,894	5,188
14	4,022	2,453	2,383	5,326	4,938	5,064
15	2,239	2,669	2,375	5,091	5,008	4,968
16	2,070	2,598	2,566	5,005	5,023	5,006
17	2,571	2,728	2,886	4,938	4,949	4,980
18	2,520	2,440	2,919	4,942	4,923	5,057
19	2,432	2,862	3,565	4,949	4,993	4,646
20	2,707	3,018	3,484	4,894	5,235	5,205
21	2,221	2,686	3,765	4,949	5,132	5,038
22	2,166	2,777	2,500	5,008	5,099	5,031
23	2,710	2,339	4,177	4,963	4,995	5,007
24	2,777	2,294	3,139	5,333	5,197	5,407
25	2,306	2,401	3,800	4,928	5,170	5,868
26	2,344	2,882	3,840	5,253	4,736	5,313
27	2,112	2,762	2,411	5,747	4,978	5,093
28	1,735	2,654	2,452	5,064	5,078	5,723
29	1,772	2,474	3,273	5,205	5,063	6,378
30	2,004	2,640	3,588	5,121	5,018	5,163
31	2,821	2,562	2,642	5,181	5,055	6,000
32	2,780	2,926	3,481	6,296	4,889	4,688
33	2,889	3,177	3,578	5,239	4,997	4,900
34	2,746	3,028	2,898	—	5,152	5,403
35	2,725	2,519	3,444	—	4,942	5,416
36	3,493	3,405	2,906	—	5,270	5,430
37	2,813	2,462	2,550	—	3,949	6,753
38	2,315	2,483	2,749	—	4,983	5,932
39	2,614	4,468	2,435	—	5,377	5,426
40	2,163	3,370	2,389	—	5,528	5,134
41	2,151	2,726	2,703	—	5,515	6,154
42	2,326	2,814	2,963	—	5,027	5,351
43	2,626	2,847	2,542	—	4,907	6,138
44	2,438	2,261	3,455	—	4,946	5,093
45	2,452	2,516	3,195	—	5,015	6,058
46	2,955	2,842	3,331	—	5,257	5,094
47	2,381	2,788	3,959	—	4,820	5,086
48	2,380	2,721	3,038	—	5,014	—
49	—	3,017	3,020	—	—	—
50	—	2,506	3,251	—	—	—
51	—	2,989	—	—	—	—
52	—	2,868	—	—	—	—
53	—	3,069	—	—	—	—
54	—	2,753	—	—	—	—

Table C.13 — Variogram values for increments S13 to S18

Lag *t*	S13	S14	S15	S16	S17	S18
0	0	0	0	0	0	0
1	0,134	0,110	0,166	0,075	0,017	0,206
2	0,218	0,131	0,169	0,067	0,024	0,223
3	0,192	0,107	0,169	0,055	0,026	0,246
4	0,222	0,143	0,232	0,069	0,025	0,202
5	0,248	0,158	0,250	0,049	0,019	0,274
6	0,252	0,123	0,240	0,071	0,026	0,222
7	0,247	0,122	0,283	0,088	0,029	0,219
8	0,239	0,164	0,328	0,067	0,028	0,184
9	0,230	0,162	0,256	0,090	0,021	0,272
10	0,235	0,155	0,307	0,094	0,019	0,302
11	0,222	0,178	0,306	0,107	0,020	0,250
12	0,192	0,148	0,263	0,107	0,017	0,199
13	0,232	0,128	0,315	0,108	0,023	0,255
14	0,278	0,143	0,318	0,119	0,023	0,253
15	0,298	0,166	0,331	0,118	0,021	0,288
16	0,330	0,126	0,345	0,127	0,016	0,238
17	0,302	0,119	0,341	0,106	0,019	0,290
18	0,252	0,141	0,347	0,084	0,022	0,362

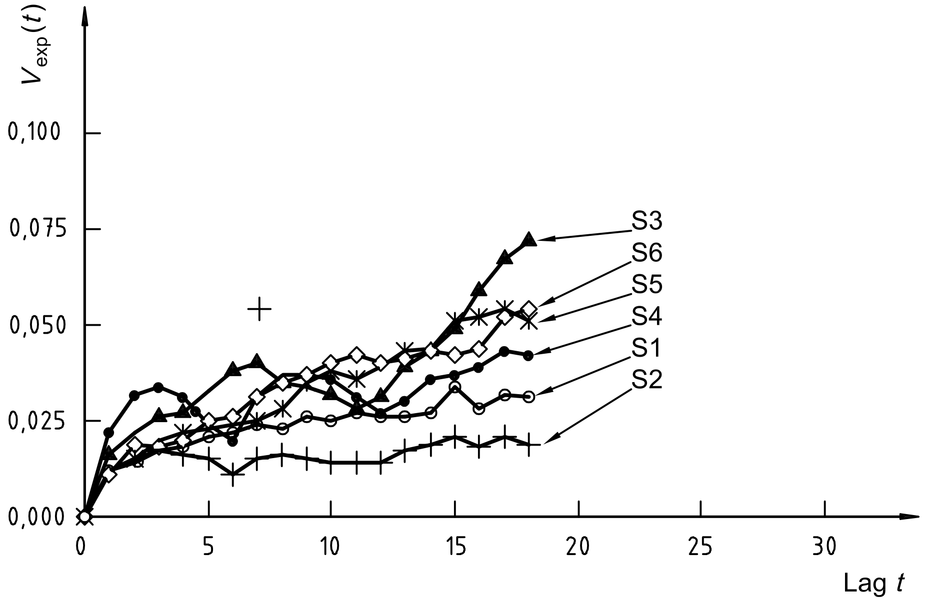


Figure C.15 — Variogram for increments S1 to S6

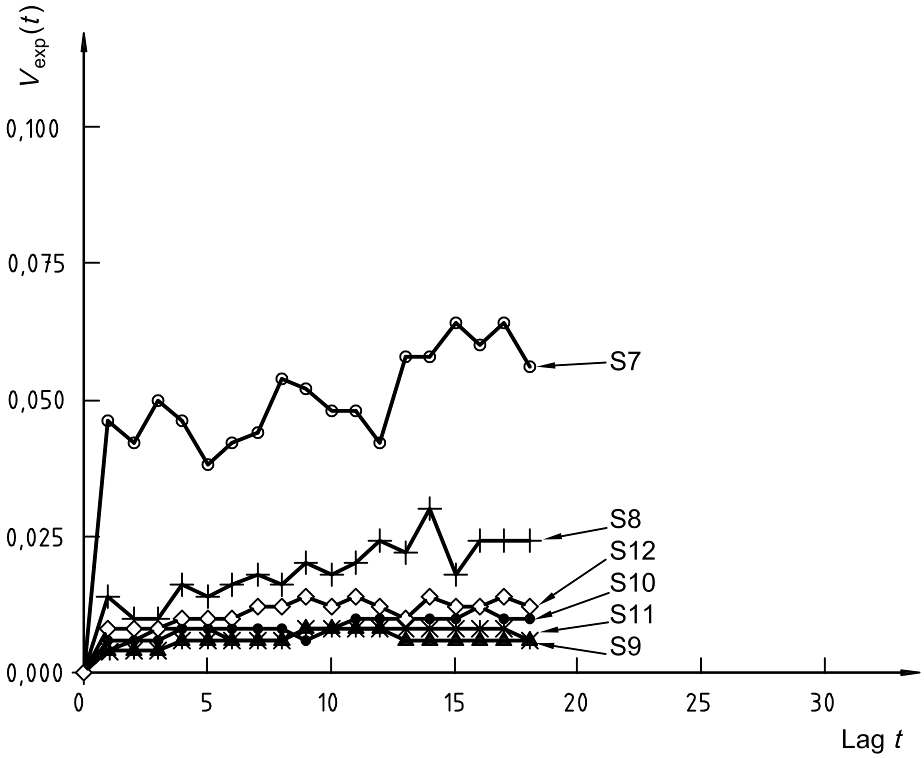


Figure C.16 — Variogram for increments S7 to S12

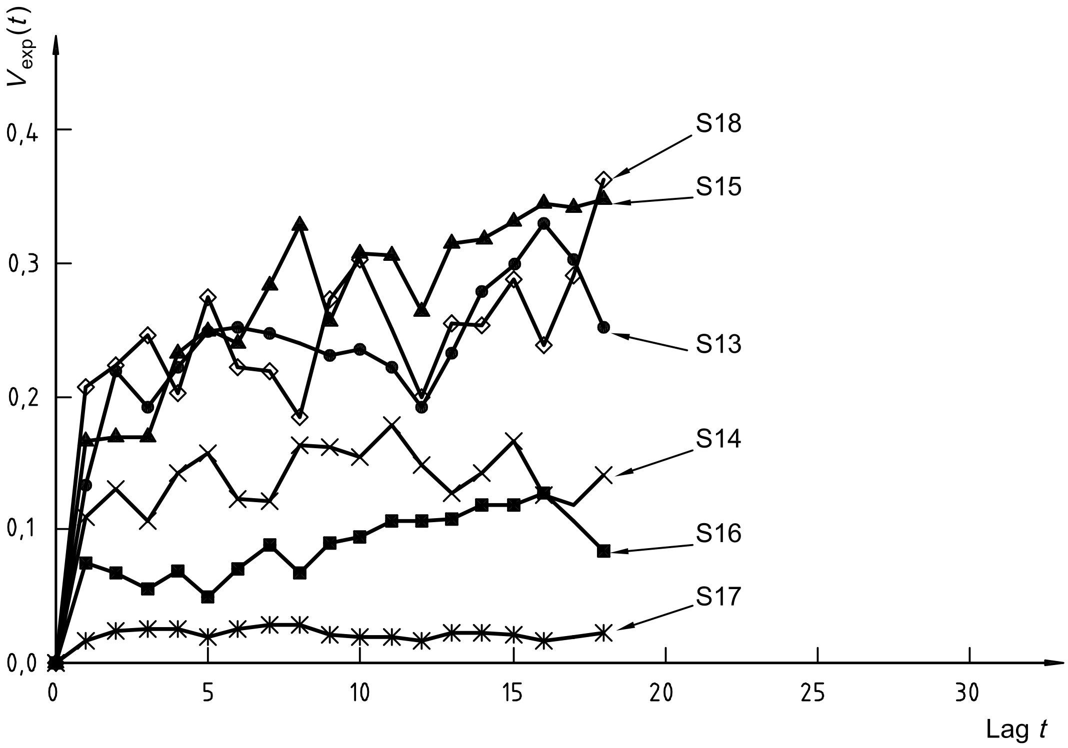


Figure C.17 — Variogram for increments S13 to S18

C.6 Size distribution and moisture content in iron ores

In C.5, a decrease in moisture with an increasing increment number of iron ores is observed in most cases with especially sharp decreases in lump ores. In order to find out the cause of these trends, paired data of moisture content and size distribution of six vessels loaded with lump ores of the same variety are collected. In this survey, increments are taken at half of the routine interval by systematic sampling on a mass basis. Odd numbered increments are used for the determination of the moisture content, while even numbered increments are used for the determination of the size distribution. Obtained data are shown in Table C.14, where w_{m} denotes moisture, expressed as a percentage by mass, and w_{sf} denotes the size distribution (fraction of iron ore with particle sizes below 6 mm).

Regressions of the moisture content and the size distribution for the increment number as well as regressions of moisture content for size distribution are analysed. Size distributions are expressed as the fraction of iron ore with particle sizes below 6 mm. Results of statistical analysis on these series are summarized in Table C.15. Variogram values are calculated on moisture content and size distribution, as shown in Tables C.16 and C.17. Variograms of moisture content and size distribution are shown in Figures C.18 and C.19, respectively.

Table C.14 — Moisture content (%) and size distribution (% fraction < 6 mm) for increments S19 to S24

No.	S19		S20		S21		S22		S23		S24	
	w_m	w_{sf}	w_m	w_{sf}	w_m	w_{sf}	w_m	w_{sf}	w_m	w_{sf}	w_m	w_{sf}
1	2,573	12,606	3,123	2,863	11,965	2,863	2,586	18,235	2,430	21,843	2,74	9,85
2	2,532	14,046	3,040	2,704	12,244	2,704	2,521	13,347	1,936	25,294	2,76	11,30
3	2,394	12,500	2,870	2,610	11,173	2,610	2,723	15,169	2,384	11,764	2,88	10,72
4	2,679	9,594	2,914	2,681	12,796	2,681	2,517	13,773	2,152	9,973	2,99	15,90
5	2,567	6,315	3,188	2,604	10,887	2,604	2,377	12,361	2,466	7,455	2,76	12,06
6	2,174	7,331	2,871	2,497	6,934	2,497	2,588	18,433	2,444	8,806	2,81	11,59
7	2,421	6,681	2,934	2,581	8,928	2,581	2,491	13,191	2,443	8,707	2,80	16,03
8	2,350	10,728	2,515	2,570	9,090	2,570	2,365	14,143	2,307	6,117	2,95	13,87
9	2,381	8,823	2,705	2,776	10,114	2,776	2,398	12,828	2,294	18,449	2,94	15,18
10	2,557	9,146	2,755	2,739	11,567	2,739	2,651	6,692	2,558	6,020	3,02	13,32
11	2,373	10,697	2,975	2,548	6,933	2,548	2,403	18,534	1,965	13,192	3,01	14,45
12	2,417	12,078	2,921	2,638	8,146	2,638	2,525	15,957	2,247	22,743	2,77	15,34
13	2,449	11,397	2,918	2,622	14,598	2,622	2,502	14,470	1,813	7,853	2,72	12,33
14	2,528	8,872	2,730	2,613	6,470	2,613	2,230	6,231	2,002	6,084	3,33	13,52
15	2,394	10,272	3,631	2,437	4,910	2,437	2,262	11,548	2,107	10,810	2,80	12,03
16	2,297	8,085	2,798	2,471	3,448	2,471	2,588	10,516	2,116	9,023	2,60	13,27
17	2,306	10,119	2,841	2,851	9,819	2,851	2,469	7,536	2,071	17,785	2,64	7,67
18	2,447	4,680	2,812	2,544	7,784	2,544	2,348	14,114	1,989	10,424	2,60	11,97
19	2,379	5,128	2,639	2,564	10,204	2,564	2,332	5,090	1,913	5,741	2,55	9,00
20	2,316	10,714	2,495	2,656	9,202	2,656	2,165	10,762	2,062	6,862	2,57	10,43
21	2,242	11,974	2,646	2,589	6,115	2,589	2,232	4,815	2,155	6,854	2,51	10,67
22	2,310	6,896	2,447	2,670	7,391	2,670	2,237	9,333	2,141	6,034	2,58	8,88
23	2,378	7,162	2,484	2,566	5,042	2,566	2,046	16,949	2,378	9,795	2,53	8,39
24	2,331	5,023	2,528	2,506	8,043	2,506	2,548	8,385	2,432	4,954	2,49	7,62
25	2,342	5,544	2,874	2,610	8,695	2,610	2,340	7,575	1,901	6,637	2,61	6,95
26	2,641	4,516	2,542	2,765	16,560	2,765	2,722	4,469	1,939	7,848	2,60	17,97
27	2,394	4,871	2,504	2,441	11,326	2,441	2,326	7,079	1,826	8,787	2,63	9,09
28	2,438	3,614	2,603	2,397	5,389	2,397	2,290	3,562	1,809	7,692	2,76	6,56
29	2,395	7,908	2,395	2,931	2,764	2,931	2,313	12,121	1,729	6,591	2,72	7,78
30	2,330	7,808	2,109	2,896	4,304	2,896	2,588	15,300	1,680	8,158	2,57	6,74
31	2,392	3,719	2,412	3,871	6,578	3,871	2,459	13,533	2,156	5,384	2,50	5,49
32	2,174	5,291	2,426	2,823	3,484	2,823	2,674	7,077	1,947	12,703	2,32	5,10
33	2,386	3,977	2,398	3,432	2,380	3,432	2,547	7,155	1,648	6,230	2,46	9,63
34	2,180	6,788	2,365	2,611	6,578	2,611	2,517	14,742	1,386	5,276	2,43	6,68
35	2,276	2,912	2,559	3,498	0,800	3,498	2,556	7,730	1,895	19,458	2,35	7,37
36	2,179	2,836	2,697	3,245	2,222	3,245	2,403	6,796	1,788	7,950	2,46	7,16
37	2,398	2,811	2,711	3,588	2,727	3,588	—	—	1,699	4,359	2,59	6,86
38	2,339	4,699	2,591	4,574	11,818	4,574	—	—	1,695	4,249	2,21	9,42
39	2,507	3,676	2,734	2,917	16,772	2,917	—	—	1,913	5,357	2,30	8,45
40	2,281	6,527	2,351	2,540	1,785	2,540	—	—	2,249	7,762	2,27	5,79
41	2,144	4,407	2,551	2,598	2,659	2,598	—	—	2,101	7,829	2,45	4,99
42	2,190	2,739	2,397	2,174	3,278	2,174	—	—	1,813	5,555	2,37	6,47
43	1,999	4,000	2,269	2,502	9,696	2,502	—	—	—	—	2,12	6,26
44	2,201	2,447	2,510	2,539	7,446	2,539	—	—	—	—	2,17	9,42
45	2,265	6,229	2,409	2,334	6,341	2,334	—	—	—	—	2,16	7,49
46	2,203	10,666	—	2,378	6,748	2,378	—	—	—	—	1,98	8,62
47	1,984	3,902	—	2,640	5,056	2,640	—	—	—	—	—	—
48	2,239	4,332	—	2,849	3,401	2,849	—	—	—	—	—	—
49	2,429	11,337	—	2,533	4,511	2,533	—	—	—	—	—	—
50	2,280	13,151	—	2,475	4,761	2,475	—	—	—	—	—	—
51	1,977	4,310	—	2,441	1,785	2,441	—	—	—	—	—	—
52	2,141	3,954	—	3,120	0,626	3,120	—	—	—	—	—	—
53	2,012	7,051	—	—	—	—	—	—	—	—	—	—
54	2,569	2,564	—	—	—	—	—	—	—	—	—	—

Table C.15 — Summary of statistical analysis for increments S19 to S24

Series No.	Number of increment	Outlier		Regressions			V_{exp} (t = 1)	
		w_{m}	w_{sf}	$w_{\text{m}} - i$	$w_{\text{sf}} - i$	$w_{\text{m}} - w_{\text{sf}}$	w_{m}	w_{sf}
S19	54	0	0	—[a]	—[a]	+[b]	0,016	4,622
S20	45	1	0	—[a]	—[b]	0	0,034	7,142
S21	52	1	0	0	—[a]	0	0,096	8,273
S22	36	0	0	0	—[a]	0	0,020	14,289
S23	42	0	1	—[a]	—[a]	0	0,033	19,242
S24	46	0	0	—[a]	—[a]	+[a]	0,015	4,762

w_{m} is the mass fraction, expressed as a percentage (%), of moisture;
w_{sf} is the fraction of iron ore with particle sizes below 6 mm.
i is the increment number.

[a] Highly significant correlation.

[b] Significant correlation.

Table C.16 — Variogram values of moisture content for increments S19 to S24

Lag t	S19	S20	S21	S22	S23	S24
0	0	0	0	0	0	0
1	0,016	0,034	0,096	0,020	0,033	0,015
2	0,017	0,034	0,105	0,020	0,038	0,021
3	0,019	0,040	0,142	0,024	0,048	0,020
4	0,016	0,044	0,162	0,025	0,044	0,022
5	0,015	0,060	0,155	0,026	0,049	0,026
6	0,016	0,056	0,175	0,020	0,061	0,028
7	0,019	0,067	0,175	0,025	0,055	0,034
8	0,017	0,055	0,210	0,025	0,056	0,035
9	0,014	0,059	0,221	0,026	0,048	0,045
10	0,015	0,037	0,243	0,033	0,071	0,043
11	0,016	0,054	0,254	0,038	0,065	0,041
12	0,018	0,059	0,245	0,041	0,066	0,048
13	0,018	0,059	0,254	0,034	0,066	0,051
14	0,021	0,065	0,238	0,025	0,058	0,046
15	0,019	0,085	0,200	0,030	0,059	0,054
16	0,020	0,076	0,185	0,028	0,054	0,060
17	0,022	0,089	0,175	0,036	0,057	0,065
18	0,024	0,091	0,182	0,034	0,075	0,069

Table C.17 — Variogram values of size distribution for increments S19 to S24

Lag *t*	S19	S20	S21	S22	S23	S24
0	0	0	0	0	0	0
1	4,622	7,142	8,273	14,289	19,242	4,762
2	7,297	7,257	13,739	15,689	23,312	4,980
3	6,693	8,041	15,001	16,430	19,360	5,167
4	6,414	10,029	12,404	16,357	26,790	5,376
5	7,178	8,421	11,341	12,479	20,857	5,538
6	7,091	9,407	13,438	17,028	22,840	6,375
7	6,940	7,722	12,358	10,500	25,784	5,863
8	6,726	6,227	13,612	18,821	19,231	6,733
9	5,264	7,545	16,007	18,471	21,019	7,806
10	6,394	9,316	17,775	17,994	17,724	7,881
11	7,342	8,996	16,070	12,758	19,784	7,996
12	7,345	9,156	12,561	14,314	24,406	7,706
13	8,150	9,736	12,954	26,553	24,739	9,732
14	9,781	10,081	14,041	18,035	20,668	9,973
15	10,185	8,542	13,968	24,037	17,418	11,249
16	10,100	6,340	12,958	21,422	21,631	12,257
17	9,494	4,824	10,605	19,795	25,140	11,569
18	9,945	7,389	12,154	20,286	23,557	12,213

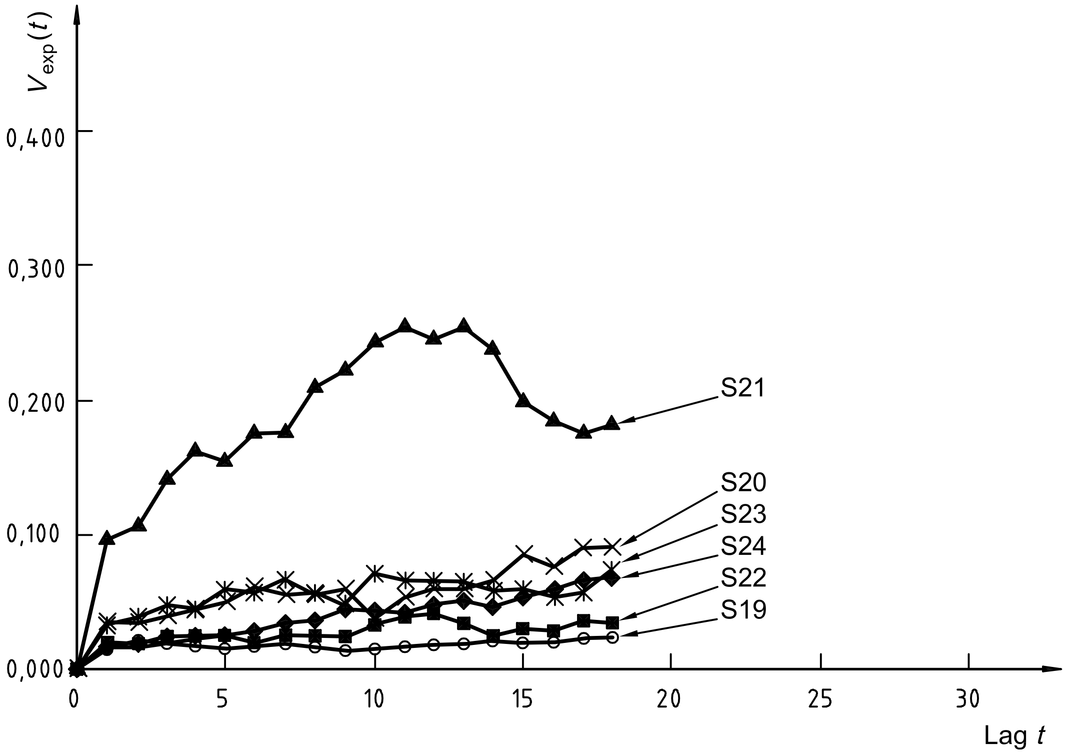


Figure C.18 — Variogram of moisture content for increments S19 to S24

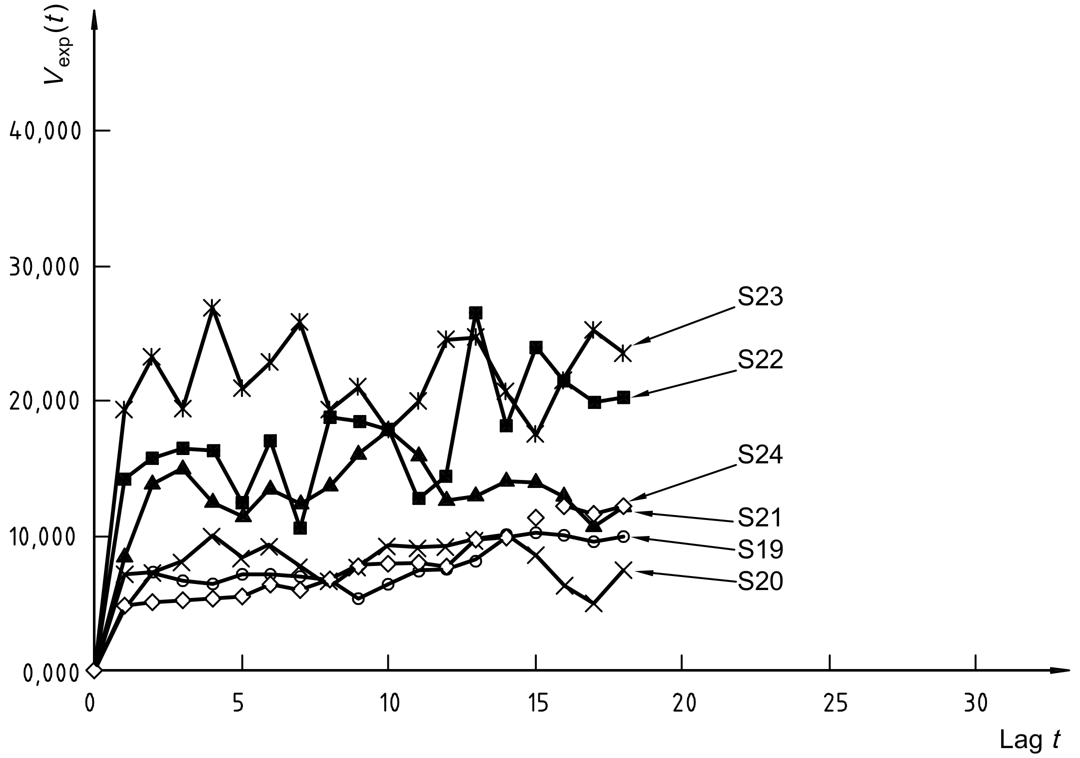


Figure C.19 — Variogram of size distribution for increments S19 to S24

C.7 Trends in iron ores

C.7.1 Collection of data

For iron ores, quality variations within strata determined by taking two increments from each stratum are checked periodically by mass-basis systematic sampling in accordance with ISO 3084 (see reference [16] of Bibliography). The survey method is interpenetrating sampling as described in 7.3. Quality variations on the same variety of iron ore as in C.5 and C.6 imported over a recent decade are collected and summarized from the survey. The format of the original data is shown in Table 5 in 7.3 and repeated for convenience purposes in Table C.18. Data collected for lumps and fines are summarized in Table C.19.

Table C.18 — An example of interpenetrating sampling

Total iron content				
No. of part	*A*	*B*	**Average**	**Range**
1	65,37	64,36	64,865	1,01
2	64,82	64,82	64,82	0
3	64,81	65,10	64,955	0,29
4	64,96	65,06	65,01	0,10
5	65,23	65,20	65,215	0,03
6	65,34	65,22	65,28	0,12
7	65,54	65,80	65,67	0,26
8	65,41	65,34	65,375	0,07
9	65,16	65,22	65,19	0,06
10	65,34	65,69	65,515	0,35
Average			65,19	0,23
σ_{wst}				0,35

Total iron content:

- Method: 3 × 2 × 10
- Steel mill: G
- Date:1985-05-19
- Tonnage: 97 101 t

C.7.2 Size distribution

Except for L04 for which the fraction of iron ore with particle sizes below 8 mm is determined instead of those below 6 mm used usually, L08 for which a size determination is not included and L10 for which 12 parts are included. Other lump series in Table C.19 include the same pattern of experiments for the fraction of particle sizes below 6 mm, which contains 10 parts, each of which contains two composite samples, regardless of the fact that each composite sample contains a different number of increments. Accordingly, the remaining nine lots will all be analysed together. Each data value for composite samples, x_{BC}, can be expressed by the following equation:

$$x_{\text{BC}} = \mu + f_{\text{BV}} + f_{\text{BP}} + f_{\text{BC}} \qquad \text{(C.10)}$$

where

μ is the population mean;

f_{BV} is the variation between vessels;

f_{BP} is the variation between parts in a vessel;

f_{BC} is the variation between composite samples within a part.

Table C.19 — Summary of survey of quality variations

Lot number	Mass of lot t	Applied method	Intervals t	Surveyed characteristics
L01	69 597	$2 \times 2 \times 10 = 40$	1 740	w_{sf}, w_{Fe}
L02	74 141	$5 \times 2 \times 10 = 100$	741	w_{sf}, w_{Fe}
L03	77 749	$5 \times 2 \times 10 = 100$	777	w_{sf}, w_{Fe}
L04	60 820	$3 \times 2 \times 10 = 60$	1 014	w_{sf}, w_{Fe}
L05	144 583	$3 \times 2 \times 10 = 60$	2 410	w_{sf}, w_{m}, w_{Fe}
L06	135 626	$4 \times 2 \times 10 = 80$	1 695	w_{sf}, w_{m}, w_{Fe}
L07	93 884	$3 \times 2 \times 10 = 60$	1 565	w_{sf}, w_{m}, w_{Fe}
L08	78 334	$3 \times 2 \times 10 = 60$	1 306	w_{Fe}
L09	97 101	$3 \times 2 \times 10 = 60$	1 618	w_{sf}, w_{Fe}
L10	50 532	$4 \times 2 \times 2 = 96$	526	w_{sf}, w_{m}, w_{Fe}
L11	111 132	$3 \times 2 \times 10 = 60$	1 852	w_{sf}, w_{m}, w_{Fe}
L12	90 892	$5 \times 2 \times 10 = 100$	909	w_{sf}, w_{m}, w_{Fe}
f01	149 081	$3 \times 2 \times 10 = 60$	2 485	w_{Fe}
f02	109 759	$4 \times 2 \times 10 = 80$	1 372	w_{sf}, w_{m}, w_{Fe}, w_{Si}, w_{Al}
f03	44 467	$2 \times 2 \times 10 = 40$	1 112	w_{sf}, w_{Fe}
f04	55 725	$3 \times 2 \times 10 = 60$	929	w_{Fe}
f05	120 073	$4 \times 2 \times 10 = 80$	1 501	w_{sf}, w_{m}, w_{Fe}, w_{Si}, w_{Al}
f06	103 051	$3 \times 2 \times 10 = 60$	1 718	w_{Fe}
f07	66 691	$2 \times 2 \times 10 = 40$	1 667	w_{m}, w_{Fe}
f08	103 956	$3 \times 2 \times 10 = 60$	1 733	w_{Fe}
f09	102 398	$3 \times 2 \times 10 = 60$	1 707	w_{sf}, w_{m}, w_{Fe}
f10	60 418	$4 \times 2 \times 10 = 80$	755	w_{sf}, w_{m}, w_{Fe}
f11	30 130	$4 \times 2 \times 11 = 88$	342	w_{sf}, w_{m}, w_{Fe}
f12	31 446	$2 \times 2 \times 12 = 48$	655	w_{sf}, w_{m}, w_{Fe}

L designates samples of lumps followed by the lot number;
f designates samples of fines followed by the lot number;
w_{sf} is the size distribution (fraction (%) of particle sizes $>$ 6 mm for lumps and $<$ 6 mm for fines);
w_{m} is the mass fraction, expressed as a percentage (%), of moisture;
w_{Fe} is the mass fraction, expressed as a percentage (%), of total iron content;
w_{Si} is the mass fraction, expressed as a percentage (%), of SiO_2;
w_{Al} is the mass fraction, expressed as a percentage (%), of Al_2O_3.

In this case, the number of vessels is nine, the number of parts is 10 and the number of composite samples is two. An ANOVA table is shown in Table C.20.

Table C.20 — ANOVA on survey data of quality variation

Sources	Sums of squares	Degrees of freedom	Mean squares	Expected mean squares
Vessels	S_{BV}	$v - 1$	$S_{\mathrm{BV}}/f_{\mathrm{BV}}$	$\sigma_{\mathrm{BC}}^2 + c\sigma_{\mathrm{BP}}^2 + pc\sigma_{\mathrm{BV}}^2$
Parts	S_{BP}	$v(p - 1)$	$S_{\mathrm{BP}}/f_{\mathrm{BP}}$	$\sigma_{\mathrm{BC}}^2 + c\sigma_{\mathrm{BP}}^2$
Composite samples	S_{BC}	$vp(c - 1)$	$S_{\mathrm{BC}}/f_{\mathrm{BC}}$	σ_{BC}^2
Total	S_{t}	$vpc - 1$		

Table C.21 is obtained by substituting numerical values.

Table C.21 — ANOVA on size distribution of lumps

Sources	Sums of squares	Degrees of freedom	Mean squares	Expected mean squares
Vessels	572,14	8	71,52	$\sigma_{\mathrm{BC}}^2 + 2\sigma_{\mathrm{BP}}^2 + 20\sigma_{\mathrm{BV}}^2$
Parts	1 289,59	81	15,92	$\sigma_{\mathrm{BC}}^2 + 2\sigma_{\mathrm{BP}}^2$
Composite samples	153,82	90	1,71	σ_{BC}^2
Total	2 015,55	179		

Equating the equations in the column of the expected mean squares to observed variances, respectively, gives

$$\hat{\sigma}_{\mathrm{BC}}^2 + 2\hat{\sigma}_{\mathrm{BP}}^2 + 20\hat{\sigma}_{\mathrm{BV}}^2 = 71{,}52$$

$$\hat{\sigma}_{\mathrm{BC}}^2 + 2\hat{\sigma}_{\mathrm{BP}}^2 = 15{,}92$$

$$\hat{\sigma}_{\mathrm{BC}}^2 = 1{,}71$$

then,

$$\hat{\sigma}_{\mathrm{BC}}^2 = 1{,}71$$

$$\hat{\sigma}_{\mathrm{BP}}^2 = 7{,}11$$

$$\hat{\sigma}_{\mathrm{BV}}^2 = 2{,}78$$

are obtained and it is observed that the variation between the parts in a vessel is the largest variance component.

Results of ANOVA on the fines data of five vessels including f02, f03, f05, f09 and f10 are shown in Table C.22.

Table C.22 — ANOVA on size distribution of fines

Sources	Sums of squares	Degrees of freedom	Mean squares	Expected mean squares
Vessels	462,65	4	115,66	$\sigma_{\text{BC}}^2 + 2\sigma_{\text{BP}}^2 + 20\sigma_{\text{BV}}^2$
Parts	142,83	45	3,17	$\sigma_{\text{BC}}^2 + 2\sigma_{\text{BP}}^2$
Composite samples	52,59	50	1,05	σ_{BC}^2
Total	658,07	99		

The variance components are estimated as follows:

$$\hat{\sigma}_{\text{BC}}^2 = 1{,}05$$

$$\hat{\sigma}_{\text{BP}}^2 = 1{,}06$$

$$\hat{\sigma}_{\text{BV}}^2 = 5{,}62$$

The between-vessels variance component is the largest.

The regression of the size distribution on the unloaded ratio is calculated for each vessel. The unloaded ratio is expressed as the ratio of the number of parts to an individual part relative to the total parts. Suppose the total parts equal to one, the slope of the regression indicates the difference between the fraction percentage at the start of unloading and that at the end of unloading. Slopes of regression of size distribution are shown in Table C.23. In Table C.23, the variances, $\hat{\sigma}_{\text{BC}}^2$ and $\hat{\sigma}_{\text{BP}}^2$, obtained by ANOVA for each vessel are shown.

Table C.23 — Slopes of regression for size distribution

Lot No.	Slope	$\hat{\sigma}_{BC}^{2}$	$\hat{\sigma}_{BP}^{2}$
L01	−11,895[a]	3,122	13,19
L02	−7,058[a]	1,936	4,57
L03	−4,150[a]	0,717	2,30
L05	−11,468[a]	1,996	12,29
L06	−6,684[a]	0,742	5,00
L07	−9,373[a]	2,205	8,88
L09	−6,371[a]	2,878	8,54
L10	−9,924[a]	0,879	9,83
L11	−7,098[a]	0,547	4,63
L12	−5,318[a]	1,239	4,56
f02	+1,71[a]	0,437	0,159
f03	+2,69	0,599	3,314
f05	+1,14	0,647	0,428
f09	−0,06	2,494	0,992
f10	−1,86[b]	1,082	0,413
f11	+ 1,55	1,911	0,250
f12	+1,85[a]	0,271	0,551

[a] Highly significant correlation.

[b] Significant correlation.

C.7.3 Moisture

Results of ANOVA on moisture of lumps, including L05, L06, L07, L11 and L12, are shown in Table C.24.

Table C.24 — ANOVA on moisture of lumps

Sources	Sums of squares	Degrees of freedom	Mean squares	Expected mean squares
Vessels	0,44	4	0,11	$\sigma_{BC}^{2}+2\sigma_{BP}^{2}+20\sigma_{BV}^{2}$
Parts	5,91	45	0,13	$\sigma_{BC}^{2}+2\sigma_{BP}^{2}$
Composite samples	1,20	50	0,02	σ_{BC}^{2}
Total	7,55	99		

Equating the equations in the column of expected mean squares to observed variances, variance components are estimated as follows:

$$\hat{\sigma}_{BC}^{2}=0{,}02$$

$$\hat{\sigma}_{BP}^{2}=0{,}055$$

$$\hat{\sigma}_{BV}^{2}=0$$

The variation between the parts in a vessel is the largest variance component.

Results of ANOVA on moisture of fines, including f02, f05, f07, f09 and f10, are shown in Table C.25.

Table C.25 — ANOVA on moisture of fines

Sources	Sums of squares	Degrees of freedom	Mean squares	Expected mean squares
Vessels	3,43	4	0,86	$\sigma_{\text{BC}}^2 + 2\sigma_{\text{BP}}^2 + 20\sigma_{\text{BV}}^2$
Parts	0,66	45	0,01	$\sigma_{\text{BC}}^2 + 2\sigma_{\text{BP}}^2$
Composite samples	0,34	50	0,01	σ_{BC}^2
Total	4,43	99		

The variance components are estimated as follows:

$$\hat{\sigma}_{\text{BC}}^2 = 0{,}01$$

$$\hat{\sigma}_{\text{BP}}^2 = 0$$

$$\hat{\sigma}_{\text{BV}}^2 = 0{,}04$$

The variation between vessels is the largest variance component.

The regression slopes, $\hat{\sigma}_{\text{BC}}^2$ and $\hat{\sigma}_{\text{BP}}^2$ of moisture are shown in Table C.26.

Table C.26 — Slopes of regression for moisture

Lot No.	Slope	$\hat{\sigma}_{\text{BC}}^2$	$\hat{\sigma}_{\text{BP}}^2$
L05	+0,061	0,074	0,019
L06	−0,181	0,004	0,012
L07	−0,455[a]	0,007	0,050
L10	+0,032	0,006	0,011
L11	+0,731[a]	0,032	0,104
L12	−0,915[a]	0,003	0,084
f02	+0,022	0,004	0,006
f05	+0,076	0,006	0
f07	−0,336[a]	0,011	0,017
f09	+0,030	0,004	0
f10	+0,011	0,009	0
f11	+0,110	0,025	0,278
f12	−0,287[a]	0,003	0,010

[a] Highly significant correlation.

C.7.4 Total iron content

Results of ANOVA for the total iron content of 10 vessels of lumps, excluding L04 (regarded as different type of ore) and L10 (different number of parts), are shown in Table C.27.

Table C.27 — ANOVA on total iron content of lumps

Sources	Sums of squares	Degrees of freedom	Mean squares	Expected mean squares
Vessels	21,98	9	2,44	$\sigma_{BC}^2 + 2\sigma_{BP}^2 + 20\sigma_{BV}^2$
Parts	9,89	90	0,11	$\sigma_{BC}^2 + 2\sigma_{BP}^2$
Composite samples	8,43	100	0,08	σ_{BC}^2
Total	40,30	199		

The estimate of the variance components are:

$$\hat{\sigma}_{BC}^2 = 0{,}08$$

$$\hat{\sigma}_{BP}^2 = 0{,}015$$

$$\hat{\sigma}_{BV}^2 = 0{,}116\,5$$

The variation between vessels is the largest variance component.

The ANOVA results of on total iron content of 10 vessels of fines, excluding f11 and f12 (both have a different number of parts), are shown in Table C.28.

Table C.28 — ANOVA on total iron content of fines

Sources	Sums of squares	Degrees of freedom	Mean squares	Expected mean squares
Vessels	25,20	9	2,80	$\sigma_{BC}^2 + 2\sigma_{BP}^2 + 20\sigma_{BV}^2$
Parts	5,71	90	0,06	$\sigma_{BC}^2 + 2\sigma_{BP}^2$
Composite samples	2,70	100	0,03	σ_{BC}^2
Total	33,61	199		

The estimate of the variance components are:

$$\hat{\sigma}_{BC}^2 = 0{,}03$$

$$\hat{\sigma}_{BP}^2 = 0{,}015$$

$$\hat{\sigma}_{BV}^2 = 0{,}137$$

The variation between the vessels is the largest variance component.

The regression slopes of total iron content are shown in Table C.29. The data for L07 and L11 are from the same steel mill D and show negative regressions. Bulldozers are usually used at the latest stage for unloading a vessel for quick discharge of iron ore. Mill D was suspected of using bulldozers at an earlier stage for unloading, thus resulting in smaller particles being segregated to the top part of the ore pile in the hold of the vessel at loading and then became mixed with the remainder of the pile.

Table C.29 — Slopes of regression of total iron content

Lot No.	Slopes	$\hat{\sigma}_{\text{BC}}^2$	$\hat{\sigma}_{\text{BP}}^2$
L01	+ 0,455	0,116	0,004
L02	+ 0,658[a]	0,040	0,042
L03	+ 0,344	0,073	0,044
L04	+ 0,868[a]	0,005	0,083
L05	+ 0,573	0,289	0
L06	+ 0,266[a]	0,013	0,002
L07	− 0,140	0,083	0
L08	+ 0,849[a]	0,098	0,033
L09	+ 0,763[a]	0,066	0,047
L10	+ 0,066	0,048	0
L11	− 0,231	0,044	0,002
L12	− 0,102	0,020	0,019
f01	+ 0,395[b]	0,023	0,039
f02	− 0,226[b]	0,021	0,003
f03	+ 0,432	0,070	0,062
f04	− 0,128	0,007	0,002
f05	− 0,122	0,014	0
f06	− 0,441[a]	0,019	0,029
f07	− 0,045	0,022	0,003
f08	− 0,249	0,041	0,026
f09	+ 0,050	0,023	0,029
f10	+ 0,050	0,030	0
f11	− 0,360[a]	0,018	0,009
f12	+ 0,113	0,022	0

[a] Highly significant correlation.

[b] Significant correlation.

C.7.5 Bias in sampling of iron ore

Trends of size distribution (fraction less than 6 mm fraction) are estimated as minus 12 % at maximum for lumps, and minus 2 % for the fraction of fines less than 6 mm (in Table C.23, about plus 2 % for the fraction greater than 6 mm).

Trends of moisture are not clear from Table C.26. However, from Table C.9 they are estimated to be, at least, less than 1,0 % for lumps and less than 0,3 % for fines.

Trends of total iron content are estimated as less than plus 1,0 % for lumps from Table C.29, while any tendency cannot be observed for fines from the same table.

In the case of maximum trend, i.e. minus 12 % of size distribution for lumps, a possible bias introduced by mass-basis systematic sampling of 50 increments will be $12/(2 \times 50) = 0{,}12$ % for fraction less than 6 mm. Similarly, a possible bias for moisture and total iron content will be $1{,}0/(2 \times 50) = 0{,}01$ %.These possible biases caused by the trends are negligible compared with the overall estimation standard deviations of the average value of the quality characteristic assessed in the lot.

Annex D
(normative)

Estimating precision

D.1 General

This annex gives experimental procedures and practical applications for estimating precision achieved through routine sampling, sample preparation and measurement. The precision achieved through routine sampling, sample preparation and measurement should be checked periodically by performing duplicate sampling. When the routine sampling method (including sample preparation and measurement) is first established or changed or when a considerable time has passed since the last check, the experiment should be carried out. This experiment should preferably be included in the respective International Standard for sampling from particular bulk materials.

Routine sampling from bulk materials is frequently carried out by mass-basis systematic sampling. When mass-basis systematic sampling is applied to particulate materials, note that routine sampling is carried out according to one of the following designs:

- Design 1: A lot is subdivided into u_{lot} sub-lots, all the increments taken from each sub-lot are aggregated into u_{lot} gross samples and u_{lot} test samples are prepared and analysed separately. The weighted average of the test results by mass of sub-lots is the estimated average value of the quality characteristic assessed in the lot. When the mass of the lot is less than the stipulated mass in subdivision criteria, the lot cannot be subdivided into sub-lots. In this case, all the increments taken from the undivided lot are aggregated into a gross sample, a test sample is prepared from the gross sample and the test sample is analysed. The result of the analysis is the estimated average quality of the lot.

- Design 2: Several increments taken from a lot are aggregated into samples, test samples are prepared from the samples and analysed separately. The weighted average of the test results by the number of increments aggregated into the samples is the estimated average value of the quality characteristic assessed in the lot. Equal numbered increments are usually aggregated into the samples.

- Design 3: All the increments taken from a lot are measured increment by increment (after preparation, if necessary). The estimated average of the test results is the average quality of the lot.

Every one of the above procedures gives the estimated average value of the quality characteristic assessed in the lot, with different precision according to the design. The precision achieved can be checked by duplicate sampling.

In the case of time-basis systematic sampling, the procedures of Design 1 are allowed but the procedures of Designs 2 and 3 cannot be applied due to proportional variation of the increments to the flow rate.

D.2 General conditions

D.2.1 Number of experiments

Experiments on no less than 20 lots of the same material should preferably be carried out. When experiments on no less than 20 lots are not feasible, experiments on at least 10 lots should be carried out.

D.2.2 Number of increments and number of composite samples

The minimum number of increments required for this experiment should be twice the specified number of increments in routine sampling. For example, if the lot is subdivided into u_{lot} sub-lots, the number of increments taken from a sub-lot is n_{sub} and one gross sample is constituted in routine sampling, the minimum number of increments necessary for the experiment should be $2n_{\text{lot}}$ and two composite samples (each of them corresponding to a gross sample in routine sampling), each constituted by n_{sub} increments respectively, should be aggregated.

D.2.3 Sample preparation and measurement

The sample preparation and measurement should be carried out according to the method specified in each relevant International Standard.

D.3 Experimental procedures

In experiments using mass-basis systematic sampling, twice the number of increments as in routine sampling, i.e. $2n_{\text{sub}}$ increments, should be taken from a sub-lot at the half interval of routine sampling and two composite samples, each constituted by n_{sub} increments respectively, should be aggregated in rotation.

In experiments using simple random sampling, twice the number of increments as in routine sampling, i.e. $2n$ increments, should be taken at random from the lot and two composite samples, each constituted by n increments respectively, should be aggregated at random.

In experiments using stratified sampling, twice the number of increments as in routine sampling should be taken at random from each stratum and an equal number of increments as in routine sampling selected at random from the increments thus taken from each stratum should be aggregated into each of the two composite samples for sub-lots or lots.

In experiments using two-stage sampling, first u_{sub} primary sampling units, corresponding to same number of sampling units as in the routine sampling, should be selected at random from the lot. Then, another u_{sub} primary sampling units should be selected in the same way. The sampling units selected at the second stage may be duplicated with the sampling units selected at the first stage. Aggregate all of the increments taken from the sampling units selected at the first stage and at the second stage separately so as to obtain two composite samples for the lot.

In experiments of any type, two composite samples should be prepared and measured separately in accordance with the procedures specified in the respective International Standard.

D.4 Statistical analysis of obtained data

Irrespective of the number of duplicate data, the upper control limit, U_{CL}, and the central line of the range chart described in ISO 8258 can be applied to detect out-of-control points and to estimate the precision achieved. As far as the duplicate sampling is applied, the factor D_3 of the range chart is zero and the chart has no lower control limit. The factor D_4 of the range chart is 3,267 and the factor d_2 of the range chart is 1,128, for duplicate data. If none of the range values exceeds the corresponding U_{CL}, then the variability may be considered to be in a state of statistical control.

After duplicate data are obtained, calculate the average range, $\overline{R}$. $D_4\overline{R} = 3{,}267\overline{R}$ gives the upper control limit for the range chart. If individual ranges are below this limit, then the range chart is judged to show a state of control. When the ranges are in a state of control, $\overline{R}/d_2 = \overline{R}/1{,}128$ gives an estimate of the standard deviation within duplicate data, i.e. the precision achieved.

D.5 Practical applications

Moisture (%) and size distribution [fraction (%) of iron ore with particle sizes below 6 mm] in a variety of iron ore are measured on the $2n$ increments taken at the half interval of the routine sampling as shown in Table D.1, where n increments are measured increment by increment in routine mass-basis systematic sampling.

Table D.1 — Moisture and size distribution in duplicate samples

No. of duplicates	Moisture %		Size distribution[a] %	
1	2,573	2,532	12,606	14,046
2	2,394	2,679	12,500	9,594
3	2,567	2,174	6,315	7,331
4	2,421	2,350	6,681	10,728
5	2,381	2,557	8,823	9,146
6	2,373	2,417	10,697	12,078
7	2,449	2,528	11,397	8,872
8	2,394	2,297	10,272	8,085
9	2,306	2,447	10,119	4,680
10	2,379	2,316	5,128	10,714
11	2,242	2,310	11,974	6,896
12	2,378	2,331	7,162	5,023
13	2,342	2,641	5,544	4,516
14	2,394	2,438	4,871	3,614
15	2,395	2,330	7,908	7,808
16	2,392	2,174	3,719	5,291
17	2,386	2,180	3,977	6,788
18	2,276	2,179	2,912	2,836
19	2,398	2,339	2,811	4,699
20	2,507	2,281	3,676	6,527
21	2,144	2,190	4,407	2,739
22	1,999	2,201	4,000	2,447
23	2,265	2,203	6,229	10,666
24	1,984	2,239	3,902	4,332
25	2,429	2,280	11,337	13,151
26	1,977	2,141	4,310	3,954

[a] Fraction of iron ore with particle sizes below 6 mm.

Both control charts for the range show a state of control and the average ranges are 0,138 and 2,150, respectively. The expected standard deviations of estimates of the average value of the quality characteristic assessed in the lot through routine sampling are estimated as follows:

For moisture,

$$\hat{\sigma}_{\text{wst}} = 0{,}138/1{,}128 = 0{,}122$$

$$\hat{\sigma}_E^2 = \hat{\sigma}_{wst}^2/26 = 0{,}000\,572\,4$$

$$\hat{\sigma}_E = 0{,}024$$

where

$\hat{\sigma}_{wst}$ is standard deviation within strata of duplicate data;

$\hat{\sigma}_E$ is standard deviation of estimates of the average moisture in the lot.

For size distribution,

$$\hat{\sigma}_{wst} = 2{,}150/1{,}128 = 1{,}906$$

$$\hat{\sigma}_E^2 = \hat{\sigma}_{wst}^2/26 = 0{,}140$$

$$\hat{\sigma}_E = 0{,}374$$

In another application for iron ore, a lot is subdivided into 10 sub-lots and five increments are taken from each sub-lot by mass-basis systematic sampling during routine sampling. For estimating precision, 10 increments are taken from each sub-lot at the half interval of the routine sampling by mass-basis systematic sampling and aggregated into two composite samples in rotation. Each composite sample is prepared separately and the total iron content is analysed. The results are shown in Table D.2.

Table D.2 — Total iron content for estimating precision

No. of sub-lot	Total iron content %		R_i
1	65,17	65,54	0,37
2	65,19	65,42	0,23
3	65,23	65,38	0,15
4	65,58	65,63	0,05
5	65,33	65,51	0,18
6	65,38	65,59	0,21
7	65,48	65,23	0,25
8	65,80	65,57	0,23
9	65,18	65,13	0,05
10	65,14	65,16	0,02
Total			1,74

The upper control limit of the range chart is: $D_4\overline{R} = 3{,}267 \times 0{,}174 = 0{,}568$. The range chart indicates a state of statistical control. The expected standard deviation of the estimate of the average value of the quality characteristic assessed in the lot through the routine sampling is estimated as follows:

$$\hat{\sigma}_{wst} = \overline{R}/d_2 = 0{,}174/1{,}128 = 0{,}154\,3$$

$$\hat{\sigma}_E^2 = (0{,}154\,3)^2/10 = 0{,}002\,381$$

$$\hat{\sigma}_E = 0{,}049$$

NOTE Experimental methods and numerical examples for checking the precision of sampling from iron ores are given in Reference [17] of the Bibliography.

Annex E
(normative)

Checking for bias

E.1 General

This annex gives the methods and practical applications for checking of the bias in the process of sampling of bulk materials. The methods are designed to estimate the random errors arising both within the system under test and within the reference system, in order to test bias of the system under test relative to the reference system, by means of a single experiment.

The methods are, in general, applicable to individual components of the sampling system under test including primary samplers, crushers, dividers and measurement instruments, as well as to the whole system under test.

In the case of duplicate experiments of the average value of the quality characteristic assessed in the lot or duplicate measurements at the measurement stage, the expected variance of estimates of the average value of the quality characteristic assessed in the lot or the measurement variance component can be obtained directly from the experiment. However, in the case of duplicate sampling of individual components of the sampling system, only variance components corresponding to the individual components under test can be obtained by the experiment. Accordingly, in general, the expected variance of estimates of the average value of the quality characteristic assessed in the lot through the routine sampling, sample preparation and measurement should be obtained by the methods given in Clause 8 and Annex D.

Various methods for bias testing have been proposed in ISO Standards (see References [18] and [19] of the Bibliography) and discussed in many books and papers (see References [20], [21] to [26] of the Bibliography). F. E. Grubbs proposed so-called “Grubb’s estimates” in his paper[27] for estimation of the error variance of system under test, the error variance of reference and the variance between samples from the paired data. His attempts were unsuccessful as the estimated error variance of one member of the paired data frequently turned out to be negative. However, this paper suggested that such information, especially error variances of paired data, would be necessary for bias testing of the paired data. In this part of ISO 11648, a method for direct estimation of error variances using duplicate observations on the system under test and the reference respectively is proposed.

E.2 Principle

In general, bias testing of a sampling system has been based on taking a series of pairs of samples of essentially the same material or of the usually handled material; one member of each pair is sampled by the system or component under test, the other member being obtained by an inherently unbiased reference method.

In the proposed methods in this annex, two members of each set are sampled by the system or component under test, the other two members being obtained by an intrinsically unbiased reference method. Error variances of the system and the reference are estimated directly from the two members of the system and the reference, respectively. The bias testing of a sampling system is carried out based on the comparison between the averages of data of the system and the reference. The number of sets of data should be determined from experience.

E.3 Procedures for bias testing

Take a minimum of 20 sets of samples for the test, two members of each set are sampled by the system or component under test, two members being obtained by an inherently unbiased reference method. The order of taking of members in a set of samples is preferably the following:

a) the first system sample,

b) the first reference sample,

c) the second system sample,

d) the second reference sample, etc.

However, in the case of testing a mechanical sampler, the order of the first system sample, the second system sample, the first reference sample and the second reference sample may be allowed. Samples in a set should be taken as closely as possible along the belt.

For four members of each set, the measurement results are determined separately and the measurement results thus obtained are subjected to statistical analysis.

No. of set	System		Reference	
1	x_{i1}	x_{i2}	y_{i1}	y_{i2}
2	x_{i2}	x_{i2}	y_{i2}	y_{i2}
—	—	—	—	—
—	—	—	—	—
20	x_{120}	x_{220}	y_{120}	y_{220}

Figure E.1 — Schematic diagram for checking for bias

E.4 Calculations for bias testing

Denote the first and the second results on the i-th system sample x_{i1} and x_{i2}, and the first and the second results on the i-th reference sample y_{i1} and y_{i2}, respectively, where $i = 1, 2, \ldots, k$. The value of k should be determined through experience, but here for convenience a value of 20 can be used.

Calculate the following values using the equations indicated.

$$g_i = x_{i1} - x_{i2} \tag{E.1}$$

$$s_e^2(x) = \frac{\sum_{i=1}^{k} g_i^2}{2k} \tag{E.2}$$

$$h_i = y_{i1} - y_{i2} \tag{E.3}$$

$$s_e^2(y) = \frac{\sum_{i=1}^{k} h_i^2}{2k} \tag{E.4}$$

Put the larger of $s_e^2(x), s_e^2(y)$ in the numerator of F_0 and calculate the ratio F_0 of the error variances,

$$F_0 = s_e^2(x)/s_e^2(y) \quad \text{or} \quad F_0 = s_e^2(y)/s_e^2(x) \tag{E.5}$$

If $F_0 > F_{\alpha/2}(\nu_1, \nu_2)$, then the null hypothesis, $s_e^2(x) = s_e^2(y)$, is rejected, and the two groups of data cannot be assumed to be drawn from populations with a common variance. The significance level α is usually set equal to 0,05, and ν_1 and ν_2 are the degrees of freedom of $s_e^2(x)$ and $s_e^2(y)$ respectively, and both are k in this case.

If $F_0 < F_{\alpha/2}(\nu_1, \nu_2)$, the two groups of data may be assumed to have a common variance.

95 % confidence limits, $T_1(x)$, $T_2(x)$ and $T_1(y)$, $T_2(y)$ are calculated as follows:

$$T_1(x), T_2(x) = \overline{\overline{x}} \pm t_{1-\alpha/2}(k) s_e(x) \tag{E.6}$$

$$T_1(y), T_2(y) = \overline{\overline{y}} \pm t_{1-\alpha/2}(k) s_e(y) \tag{E.7}$$

where

$\overline{\overline{x}}$ is the grand average of x_{i1} and x_{i2};

$\overline{\overline{y}}$ is the grand average of y_{i1} and y_{i2}.

Calculate:

$$d_i = \frac{x_{i1} + x_{i2}}{2} - \frac{y_{i1} + y_{i2}}{2} \tag{E.8}$$

$$s_d^2 = \frac{1}{k-1}\left[\sum_{i=1}^{k} d_i^2 - \frac{1}{k}\left(\sum_{i=1}^{k} d_i\right)^2\right] \tag{E.9}$$

$$A_2 = \left[\ t_{1-\alpha/2}(f)/\sqrt{k}\ \right]\ s_d \tag{E.10}$$

If $\left|\overline{d}\right| > A_2$, then the hypothesis of the equality of two means is rejected. The estimate of bias is $\overline{d}$.

E.5 Residual carbon in heavy oil

The quality characteristic is the residual carbon content after 10 % distillation in grade-A heavy oil. The reference method is a micro-distillation method specified in the National Standards and it takes about 3 h to obtain a test result. The proposed system method under test is a multi-regression equation of readings from the result of spectrophotometry and a test result can be obtained within 10 m. Experimental results are shown in Table E.1. Calculations of $s_e^2(x)$ and $s_e^2(y)$ are shown in Table E.2.

Using Equations (E.2) and (E.4), the following values are obtained:

$$s_e^2(x) = \frac{\sum_{i=1}^{k} g_i^2}{2k} = \frac{31}{40} = 0{,}775$$

$$s_e(x) = 0{,}880$$

$$s_e^2(y) = \frac{\sum_{i=1}^{k} h_i^2}{2k} = \frac{15\,830}{40} = 395{,}750$$

$$s_e(y) = 19{,}893$$

Putting $s_e^2(y)$ in the numerator of F_o,

$$F_o = s_e^2(y)/s_e^2(x) = 395{,}75/0{,}775 = 510{,}65$$

$F_{\alpha/2}(20{,}20) = 2{,}46 < F_o$. Accordingly, the two groups of data cannot be assumed to be drawn from populations with a common variance.

95 % confidence limits, $T_1(x)$, $T_2(x)$ and $T_1(y)$, $T_2(y)$ are calculated in accordance with Equations (E.6) and (E.7):

$$T_1(x), T_2(x) = \overline{\overline{x}} \pm t_{1-\alpha/2}(k)s_e(x) = \overline{\overline{x}} \pm t_{0{,}975}(20)s_e(x) = 342{,}275 \pm 2{,}086 \times 0{,}880$$
$$T_1(x) = 340{,}439, \quad T_2(x) = 344{,}111$$

$$T_1(y), T_2(y) = \overline{\overline{y}} \pm t_{1-\alpha/2}(k)s_e(y) = \overline{\overline{y}} \pm t_{0{,}975}(20)s_e(y) = 312{,}700 \pm 2{,}086 \times 19{,}893$$
$$T_1(y) = 271{,}203, \quad T_2(y) = 354{,}197$$

Calculate:

$$d_i = \frac{x_{i1} + x_{i2}}{2} - \frac{y_{i1} + y_{i2}}{2}$$

$$s_d^2 = \frac{1}{k-1}\left[\sum_{i=1}^{k} d_i^2 - \frac{1}{k}\left(\sum_{i=1}^{k} d_i\right)^2\right] = \frac{1}{19}\left[23\,486{,}25 - 591{,}5^2/20\right] = 315{,}402, \quad s_d = 17{,}760$$

$$A_2 = \left[\ t_{1-\alpha/2}(v)/\sqrt{k}\right]\ s_d = \left[2{,}093/4{,}472\right] \times 17{,}76 = 8{,}312$$

$$\left|\ \overline{d}\ \right| = 29{,}575 > A_2$$

Accordingly, the hypothesis of the equality of two means is rejected. The estimate of bias is $\overline{d} = 30$.

Table E.1 — Experimental results of residual carbon in heavy oil

Values given as percentage by mass × 10^3

Sample No.	System		Reference	
	x_{i1}	x_{i2}	y_{i1}	y_{i2}
1	335	335	318	343
2	343	342	310	307
3	340	342	356	300
4	355	357	372	329
5	346	347	318	332
6	348	346	302	320
7	356	359	325	301
8	351	353	344	351
9	343	343	326	317
10	351	351	320	315
11	315	314	296	276
12	329	329	282	283
13	334	334	280	292
14	330	330	353	317
15	331	331	261	312
16	345	345	276	300
17	345	344	280	304
18	333	334	283	317
19	359	359	313	350
20	353	354	299	328
Total	6 842	6 849	6 214	6 294
Average	342,275		312,700	

Table E.2 — Calculation of $s_e^2(x)$ and $s_e^2(y)$

Sample No.	$g_i = x_{i1} - x_{i2}$	g_i^2	$h_i = y_{i1} - y_{i2}$	h_i^2
1	0	0	–25	625
2	1	1	3	9
3	–2	4	56	3 136
4	–2	4	43	1 849
5	–1	1	–14	196
6	2	4	–18	324
7	–3	9	24	576
8	–2	4	–7	49
9	0	0	9	81
10	0	0	5	25
11	1	1	20	400
12	0	0	–1	1
13	0	0	–12	144
14	0	0	36	1 296
15	0	0	–51	2 601
16		0	–24	576
17	0	1	–24	576
18	1	1	–34	1 156
19	–1	0	–37	1 369
20	0	1	–29	841
Total		31		15 830

Nonetheless, the error variance of the proposed system is much smaller than the error variance of the reference method. It is preferable to try one more experiment, so as to make sure that between duplicate measurements of the proposed system are independent, for example by obtaining duplicate measurements of the proposed system on different days.

E.6 Filling capacity of tobacco

The filling capacity of tobacco is tested in the production of cigarettes in order to assure the hardness of cigarettes. Recently, a new filling power tester has been introduced to replace the old one. The relationship between the old and the new testers is examined. 200 g of test samples are collected from the lot of about 200 kg by the ordinary sampling method. Test portions of 15 g for the old tester and of 10 g for the new tester are taken from the test samples. Duplicate measurements by the old and the new testers on the lot are shown in Table E.3.

Table E.3 — Filling capacity of tobacco by old and new testers

Values in cubic centimetres per gram

Lot No.	Old		New	
	x_{i1}	x_{i2}	y_{i1}	y_{i2}
1	4,590	4,509	4,940	4,969
2	4,399	4,346	4,792	4,760
3	4,700	4,700	5,259	5,312
4	4,562	4,619	5,131	5,114
5	4,619	4,700	4,846	4,731
6	4,399	4,481	4,771	4,763
7	4,183	4,318	4,520	4,516
8	4,644	4,562	4,910	4,874
9	5,214	5,189	5,988	6,168
10	5,405	5,270	6,215	6,066
11	5,026	4,998	5,751	5,755
12	4,916	4,916	5,581	5,600
13	5,107	5,026	5,704	5,943
14	4,427	4,456	4,756	4,758
15	4,807	4,782	5,738	5,591
16	4,863	4,888	5,581	5,568
17	5,107	5,132	5,864	5,804
18	4,835	4,888	5,630	5,572
19	5,026	4,970	5,628	5,570
20	5,894	5,866	6,946	6,856
Total	96,723	96,616	108,551	108,290
Average	4,833		5,421	

Using Equations (E.2) and (E.4), the following values are obtained:

$$s_e^2(x) = \frac{\sum_{i=1}^{k} g_i^2}{2k} = \frac{0{,}086\ 493}{40} = 0{,}002\ 173\ 5$$

$$s_e(x) = 0{,}047$$

$$s_e^2(y) = \frac{\sum_{i=1}^{k} h_i^2}{2k} = \frac{0{,}171\ \ 873}{40} = 0{,}004\ 296\ 8$$

$$s_e(y) = 0{,}066$$

Putting $s_e^2(y)$ in the numerator of F_0,

$$F_0 = s_e^2(y)/s_e^2(x) = 0{,}004\ 296\ 8/0{,}002\ 173\ 5 = 1{,}98$$

and $F_{\alpha/2}(20{,}20) = 2{,}46 \quad > F_0$

The two groups of data may be assumed to have a common variance.

95 % confidence limits, $T_1(x)$, $T_2(x)$ and $T_1(y)$, $T_2(y)$ are calculated in accordance with Equations (E.6) and (E.7):

$$T_1(x), T_2(x) = \bar{\bar{x}} \pm t_{1-(\alpha/2)}(k)s_e(x) = \bar{\bar{x}} \pm t_{0{,}975}(20)s_e(x) = 4{,}833 \pm 2{,}086 \times 0{,}047$$

$$T_1(x) = 4{,}735, \quad T_2(x) = 4{,}931$$

$$T_1(y), T_2(y) = \bar{\bar{y}} \pm t_{1-(\alpha/2)}(k)s_e(y) = 5{,}421 \pm 2{,}086 \times 0{,}066$$

$$T_1(y) = 5{,}283, \quad T_2(y) = 5{,}559$$

Calculate:

$$d_i = \frac{x_{i1} + x_{i2}}{2} - \frac{y_{i1} + y_{i2}}{2}$$

$$s_d^2 = \frac{1}{k-1}\left[\sum_{i=1}^{k} d_i^2 - \frac{1}{k}\left(\sum_{i=1}^{k} d_i\right)^2\right] = \frac{1}{19}\left[8{,}275\ 14 - (-11{,}751)^2/20\right] = 0{,}059\ 117, \quad s_d = 0{,}243$$

$$A_2 = \left[t_{1-\alpha/2}(v)/\sqrt{k}\right]s_d = \left[2{,}093/4{,}472\right] \times 0{,}243 = 0{,}114$$

$$\left|\bar{d}\right| = 0{,}588 \quad > A_2$$

The hypothesis of the equality of two means is rejected. The estimate of bias is minus 0,588.

E.7 COD of exhaust water

The chemical oxygen demand (COD) (parameter of water quality) of exhaust water from a plant is regulated by two methods according to the location of the plant. One is regulation by total amount of substance relative to the COD, the other is regulation by the mass concentration value of COD, ρ_{COD}, of exhaust water. A chemical plant under investigation is located in the area under the regulation of the latter.

Samples are taken from a storage tank of exhaust water of the chemical plant before dilution by fresh water and CODs are measured. Dilution ratios by fresh water are determined based on COD values obtained. The standard method specified in National Standards takes about 2,5 h, whereas the rapid method takes 15 min to 20 min.

In order to compare the precision and bias between the rapid method and the standard method, CODs are determined in duplicate on the lots by the rapid method and the standard method, respectively. Second determinations of duplicate data by the rapid method are obtained on different days, ensuring the independence of the duplicate data. Obtained data are shown in Table E.4.

Table E.4 — COD of exhaust water before dilution

Values in milligrams of oxygen per litre

Sample No.	Rapid method		Standard method	
	x_{i1}	x_{i2}	y_{i1}	y_{i2}
1	3 850	4 025	4 248	4 512
2	4 050	4 300	3 902	4 513
3	3 525	3 925	3 576	4 028
4	2 575	3 400	3 004	3 491
5	4 300	4 450	4 794	5 004
6	3 250	1 825	3 357	3 174
7	2 875	2 215	2 699	2 656
8	3 725	2 975	3 634	3 482
9	3 750	4 325	4 380	4 500
10	201	229	240	229
11	37	33	45	42
12	655	655	1 928	1 838
13	1 788	1 650	2 001	1 987
14	1 675	1 215	1 555	1 476
15	2 050	2 050	2 308	2 384
16	1 180	1 200	1 764	1 740
17	2 325	1 750	2 543	2 385
18	2 100	2 588	3 022	3 040
19	2 650	2 913	2 815	2 823
20	3 050	3 163	3 526	3 230
21	2 488	2 738	2 781	2 554
22	1 340	1 345	1 517	1 554
23	2 575	2 113	2 296	1 874
24	1 800	1 520	1 802	1 838
25	2 400	2 400	2 895	2 745
26	3 150	3 488	3 708	3 945
27	2 875	2 463	3 021	2 963
28	2 413	2 388	2 570	2 555
29	1 925	1 463	2 065	1 948
30	1 975	2 488	2 795	2 861
Average	2 395,9		2 702,7	

Using Equations (E.2) and (E.4), the following values are obtained:

$$s_e^2(x) = \frac{\sum_{i=1}^{k} g_i^2}{2k} = \frac{6\,440\,646}{60} = 107\,344{,}1$$

$$s_e(x) = 327{,}6$$

$$s_e^2(y) = \frac{\sum_{i=1}^{k} h_i^2}{2k} = \frac{1\,468\,000}{60} = 24\,466{,}7$$

$$s_e(y) = 156{,}4$$

Putting $s_e^2(x)$ in the numerator of F_o,

$$F_\text{o} = s_e^2(x)/s_e^2(y) = 107\,344{,}1/24\,466{,}7 = 4{,}39$$

and $F_{\alpha/2}(30,30) = 2{,}07 \quad < F_\text{o}$.

Accordingly, the two groups of data cannot be assumed to be drawn from populations with a common variance.

The 95 % confidence limits, $T_1(x)$, $T_2(x)$ and $T_1(y)$, $T_2(y)$, are calculated in accordance with Equations (E.6) and (E.7):

$$T_1(x), T_2(x) = \bar{\bar{x}} \pm t_{1-\alpha/2}(k) s_e(x) = \bar{\bar{x}} \pm t_{0,975}(30) s_e(x) = 2\,395{,}9 \pm 2{,}042 \times 327{,}6$$

$$T_1(x) = 1\,726{,}9, \quad T_2(x) = 3\,064{,}9$$

$$T_1(y), T_2(y) = \bar{\bar{y}} \pm t_{1-\alpha/2}(k) s_e(y) = \bar{\bar{y}} \pm t_{0,975}(30) s_e(y) = 2\,702{,}7 \pm 2{,}042 \times 156{,}4$$

$$T_1(y) = 2\,383{,}3, \quad T_2(y) = 3\,022{,}1$$

Calculate:

$$d_i = \frac{x_{i1} + x_{i2}}{2} - \frac{y_{i1} + y_{i2}}{2}$$

$$s_d^2 = \frac{1}{k-1}\left[\sum_{i=1}^{k} d_i^2 - \frac{1}{k}\left(\sum_{i=1}^{k} d_i\right)^2\right] = \frac{1}{29}\left[5\,184\,108 - (-9\,204)^2/30\right] = 81\,390{,}355, \quad s_d = 285{,}3$$

$$A_2 = \left[t_{1-\alpha/2}(\nu)/\sqrt{k}\right] s_d = \left[2{,}045/5{,}477\right] \times 285{,}3 = 106{,}5$$

$$\left|\bar{d}\right| = 306{,}8 \quad > A_2$$

Accordingly, the hypothesis of the equality of two means is rejected. The estimate of bias is minus 306,8.

The urgent task of reduction of the effect of poor precision of the rapid method will be solved by increasing the number of measurements per lot, but a plan for installation of a more precise and somewhat expensive instrument providing an alternative rapid method is being considered.

E.8 Portable versus installed emission spectrophotometer

A portable emission spectrophotometer is introduced for rapid analysis of chemical components in aluminium ingot, through direct emission on the surface of ingot. The principle of the portable spectrophotometer is the same as for the installed spectrophotometer.

At first, a test of the portable spectrophotometer compared with the installed meter is investigated using the method stipulated in ISO 3301. Si and Fe are analysed, but here only Si will be dealt with. Results are shown in Table E.5, where x_{i1} and y_{i1} are the results on the i-th sample by the portable and installed spectrophotometers, respectively.

Table E.5 — Determination of Si (%) using portable and installed spectrophotometers

Sample No.	x_{i1}	y_{i1}	$d_{i1} = x_{i1} - y_{i1}$	${D_{i1}}^2$
1	1,117	0,895	0,222	0,049 284
2	3,270	2,418	0,852	0,725 904
3	0,720	0,620	0,100	0,010 000
4	2,563	2,026	0,537	0,288 369
5	1,087	0,922	0,165	0,027 225
6	2,627	1,898	0,729	0,531 441
7	0,933	0,783	0,150	0,022 500
8	1,977	1,518	0,459	0,210 681
9	0,677	0,627	0,050	0,002 500
10	2,060	1,677	0,383	0,146 689
11	0,453	0,487	−0,034	0,001 156
12	1,613	1,410	0,203	0,041 209
13	1,097	0,965	0,132	0,017 424
14	0,493	0,470	0,023	0,000 529
15	2,187	1,688	0,499	0,249 001
16	0,567	0,482	0,085	0,007 225
17	2,023	1,656	0,367	0,134 689
18	0,457	0,386	0,071	0,005 041
19	1,643	1,491	0,152	0,023 104
20	1,073	0,946	0,127	0,016 129
21	1,120	0,925	0,195	0,038 025
Total	29,757	24,290	5,467	2,548 125
Average	1,417 0	1,156 7	0,260 3	—

$$s_d^2(1) = \frac{1}{n-1}\left[\sum_{i=1}^{21} d_{i1}^2 - \frac{1}{n}\left(\sum_{i=1}^{21} d_{i1}\right)^2\right] = \frac{1}{20}\left[2{,}548\,125 - \frac{1}{21}(5{,}467)^2\right] = 0{,}056\,244$$

$$t_o = \frac{\bar{d}_1}{\sqrt{\frac{s_d^2(1)}{n}}} = \frac{0{,}260\,3}{\sqrt{\frac{0{,}056\,244}{21}}} = 5{,}030$$

since $t_{1-\alpha/2}(v) = t_{0{,}975}(20) = 2{,}086$ and the null hypothesis: $\bar{d}_1 = 0$ has been rejected. Namely, a highly significant bias between the portable spectrophotometer and the installed spectrophotometer has been concluded. However, ISO 3301 only makes mention of the comparison of two means and not for testing for bias.

After a month, an experiment which gives the intermediate precision measured on the same samples a month apart was considered, but the samples used in the above experiment had not been kept. Then, a new experiment was designed and two subsequent measurements a day apart on the same sample by the portable spectrophotometer and the installed spectrophotometer were obtained as shown in Table E.6, where x_{i2} and x_{i3} were measured using the portable spectrophotometer, and y_{i2} and y_{i3} were measured using the installed spectrophotometer.

Table E.6 — Duplicate measurements of Si (%) using portable and installed spectrophotometers

Sample No.	x_{i2}	x_{i3}	y_{i2}	y_{i3}
1	1,160	1,085	1,041	1,059
2	1,525	1,450	1,487	1,455
3	0,340	0,315	0,342	0,334
4	1,200	1,100	1,132	1,067
5	0,455	0,405	0,435	0,428
6	1,820	1,730	1,657	1,611
7	0,405	0,355	0,382	0,381
8	2,030	1,820	1,750	1,717
9	0,465	0,410	0,435	0,432
10	2,130	1,925	1,807	1,812
11	1,205	1,290	1,126	1,112
12	0,915	0,850	0,909	0,897
13	1,915	1,630	1,633	1,640
14	2,825	2,610	2,410	2,317
15	1,955	1,930	1,680	1,726
16	0,755	0,740	0,724	0,726
17	2,580	2,200	1,682	1,653
18	1,945	1,895	1,743	1,774
19	1,450	1,320	1,275	1,298
20	1,895	1,815	2,081	2,030
21	1,990	1,710	1,630	1,686
Total	30,960	28,585	27,361	27,155
Average	1,417 7		1,2980	

Using Equations (E.2) and (E.4), the following values are obtained:

$$s_e^2(x) = \frac{\sum_{i=1}^{k} g_i^2}{2k} = \frac{0{,}512\ 475}{42} = 0{,}012\ 202$$

$$s_e(x) = 0{,}110\ 5$$

$$s_e^2(y) = \frac{\sum_{i=1}^{k} h_i^2}{2k} = \frac{0{,}028\,152}{42} = 0{,}000\,670$$

$$s_e(y) = 0{,}025\,9$$

Putting $s_e^2(x)$ in the numerator of F_0,

$$F_0 = s_e^2(x)/s_e^2(y) = 0{,}122\,02/0{,}000\,670 = 18{,}21$$

and $F_{\alpha/2}(40{,}40) = 1{,}88 \quad < F_0$

Accordingly, the two groups of data cannot be assumed to be drawn from populations with a common variance.

The 95 % confidence limits, $T_1(x)$, $T_2(x)$ and $T_1(y)$, $T_2(y)$ are calculated in accordance with Equations (E.6) and (E.7):

$$T_1(x), T_2(x) = \overline{\overline{x}} \pm t_{1-\alpha/2}(k)\,s_e(x) = \overline{\overline{x}} \pm t_{0{,}975}(21)\,s_e(x) = 1{,}417\,7 \pm 2{,}080 \times 0{,}110\,5$$

$$T_1(x) = 1{,}187\,7, \quad T_2(x) = 1{,}647\,7$$

$$T_1(y), T_2(y) = \overline{\overline{y}} \pm t_{1-\alpha/2}(k)\,s_e(y) = \overline{\overline{y}} \pm t_{0{,}975}(21)\,s_e(y) = 1{,}298\,0 \pm 2{,}080 \times 0{,}025\,9$$

$$T_1(y) = 1{,}244\,1, \quad T_2(y) = 1{,}351\,9$$

Calculate:

$$d_i = \frac{x_{i2} + x_{i3}}{2} - \frac{y_{i2} + y_{i3}}{2}$$

$$s_d^2 = \frac{1}{k-1}\left[\sum_{i=1}^{k} d_i^2 - \frac{1}{k}\left(\sum_{i=1}^{k} d_i\right)^2\right] = \frac{1}{20}\left[0{,}965\,752\,75 - 2{,}514\,5^2/21\right] = 0{,}033\,23, \quad s_d = 0{,}182$$

$$A_2 = \left[t_{1-\alpha/2}(v)\sqrt{k}\right] s_d = \left[208\,6/4{,}472\right] \times 0{,}182 = 0{,}085$$

$$\left|\overline{d}\right| = 0{,}120 \quad > A_2$$

Accordingly, the hypothesis of the equality of two means is rejected. The estimate of bias is $\overline{d} = 0{,}120$.

Since confidence intervals $T_1(x)$, $T_2(x)$ and $T_1(y)$, $T_2(y)$ overlap, it is inconclusive whether or not a bias exists between the portable spectrophotometer and the installed spectrophotometer.

Every effort, such as more careful grinding of the surface of ingot to be emitted, is being devoted to the improvement of the precision of the portable spectrophotometer.

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