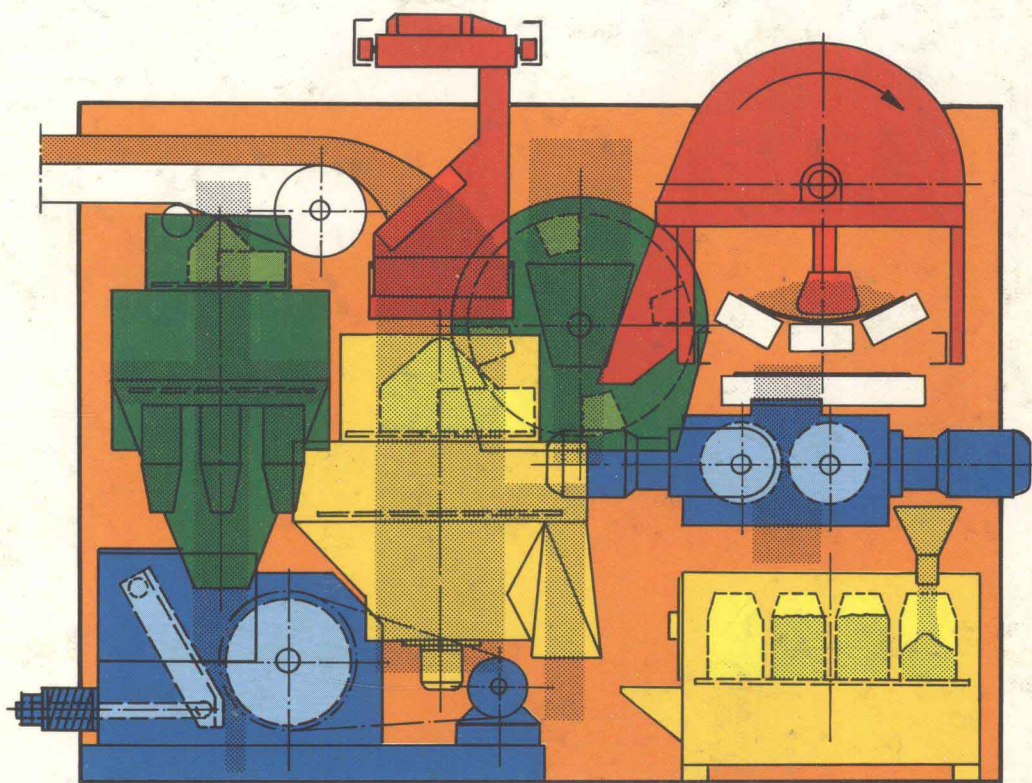


**J.W. MERKS**

# **SAMPLING AND WEIGHING OF BULK SOLIDS**



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**J. W. Merks**

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**Sampling and Weighing  
of Bulk Solids**

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# **SAMPLING AND WEIGHING OF BULK SOLIDS**

## **PREFACE**

This text is written for the specific purpose of providing guidelines in the field of quality evaluation and mass measurement, loss prevention and loss control for bulk solids such as coal and concentrates, ores and industrial minerals, chemicals and fertilizers, and many others. Its central theme is to create understanding for the concepts of accuracy and precision of measurements in weighing, sampling, preparation and analysis of bulk solids.

Generally, the accuracy of a measurement is ensured by selecting a sequence of suitable and reliable procedures, and by executing each procedure with due care and attention to ascertain that no systematic error or bias is introduced. Precision, however, comes at a price!! Hence, the cost for the determination of quality and weight must be optimized against the risk to lose. Effective loss prevention and loss control programs can only be enforced if the precision of each step in a chain of measurements is known.

In a text on sampling and weighing the principles of probability and applied statistics must be introduced for their use is essential and indispensable for the evaluation of mechanical sampling systems, manual sampling regimes, sample preparation procedures, analytical methods or weighing devices. It is not possible to discuss accuracy and precision of measurements without reference to the concept of variances. Neither is it possible to compare and evaluate measurements effectively without application of statistical tests.

Applied statistics not only provide tools and techniques to determine random variations and to detect systematic errors in samples, systems and procedures, but also permit efficient design and effective evaluation of test programs and experiments. Therefore, the most important tests and techniques will be introduced, and their use demonstrated with practical examples from a wide range of experiences and experiments at coal mines,

hard rock mines, mineral processing and coal preparation plants, steel plants and base metal smelters, power utilities and bulk handling terminals in different parts of the world.

The formulae in this text are used without derivation or proof, and the number of formulae is kept to a minimum. For those who want to learn more about the power of applied statistics, a list of applicable literature and references is included.

Efforts have also been made to eliminate statistical jargon and to use simple prose. The most important concepts, and their symbols, are defined and listed in a glossary. Many numerical examples have been included to put into perspective the basic concepts of probability and statistics, and to encourage the use of these powerful techniques. Each chapter is a separate and integral entity, but some overlap between chapters could not be avoided without loss in coherence and logical progression.

In NBS Handbook 44 on weighing, precision characteristics of weighing devices and systems are defined in terms of tolerances. In this text, the coefficient of variation is used as a measure for precision. Unless tolerances are, by definition, equal to 95 % or 99 % confidence intervals, this popular measure for the precision of a scale cannot be used to calculate how random variations in sampling, preparation of samples, analysis and weighing, propagate and impact on confidence intervals and ranges for dry weights, dry metal contents, commodity values, or any other composite parameter.

In this text I have condensed more than twenty-five years of experience and expertise in quality evaluation, mass measurement, loss prevention and loss control for a wide range of bulk solids. Surveys, seminars and workshops in different parts of the world in combination with many years of committee activities ensure that all guidelines on weighing, sampling, sample preparation and analysis are practical and useful, and that the terminology used throughout the text is in close agreement with ISO Recommendations and ASTM Standards.

The compilation of a text on these subjects is a logical sequel to my activities in various capacities with the SGS Organization, the largest independent inspection company in the world, and in the position of Assistant to the Chairman with Cominco Ltd., a large and diversified mining and smelting conglomerate with interests in zinc, lead, silver, copper, gold, potash, fertilizers and many other products.

While I was compiling this text I became even more aware that I am indebted to more people than I could possibly give credit to at this stage, to people that I worked with at some time during my career, to people

that I met during my travels, and to people who attended to my seminars and workshops. I owe them, and I thank them, because they all helped me in different ways.

For their valuable comments and criticism I am grateful to Mr. E. NIXON who is a consultant to the SGS Organization, and to Dr. D. LAGUITTON who is a Research Scientist at the Mineral Dressing Section of CANMET in Ottawa.

I owe a great deal to Mr. N. M. ANDERSON, Chairman of Cominco Ltd., and to Messrs. E. PRIFER and U. K. HAHN, Vice Presidents with SGS Control Services Inc. in New York, who encouraged me and enabled me to put it all together in this format. Last but not least I thank my wife who laboured with me while we worked through numerous drafts on our IBM Displaywriter.

I accept responsibility for errors and omissions, and I invite readers and users to submit comments and criticism so that future editions can be improved both in contents and presentation.

January 1985  
Vancouver, B.C.

J. W. MERKS





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# CHAPTER 1

## GLOSSARY

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*“When I choose a word, it means just what  
I choose it to mean — neither more nor less!”*

*“Through the Looking Glass” by Lewis Carroll*

\* \* \* \* \*

### 1.1 Definitions

#### **Accuracy**

A term used to indicate the reliability of a measurement, with a system or on a sample, in terms of the closeness of agreement between this estimate and the unknown true value.

Remark: Accuracy is a qualifying term, for example, poor accuracy, or lack of accuracy.

#### **Action Limit**

A 99% confidence range for a parameter on a control chart.

Remark: Upper and lower action limits are used in control charts for accuracy and precision

#### **Analysis Sample** (pulp sample)

Final subsample prepared from a gross sample by subsequent steps of sample division and comminution.

Remark: This analysis sample may retain some residual moisture so that a moisture determination, or additional drying, is usually required.

#### **Between-laboratory Precision of Analysis** (reproducibility)

A measure for random variations between test results determined by different laboratories in different analysis samples that were collected simultaneously from a single subsample.

**Remark:** The set of analysis samples must be collected in such a manner that the variance of the last division step is significantly smaller than the variance of analysis. If the same analysis sample is submitted to different testing laboratories, then the true variance of analysis between laboratories will be obtained.

**Bias (absolute)**

A statistically significant difference between a single measurement, or the mean of a series of measurements, and its value in a Certified Reference Material, its value measured in accordance with a recognized reference procedure, or its value measured against a Certified Weight.

**Bias (relative)**

A statistically significant mean difference between series of identifiably different measurements.

**Bias (systematic error)**

A statistically significant difference between a single measurement, or the mean of a series of measurements, and the most reliable estimate for its unknown true value.

**Calibration**

The process of comparing and adjusting measurement systems or procedures against Certified Weights, or against Certified Reference Materials.

**Calibration Hierarchy**

The chain of calibrations that traces the accuracy of a weighing device to a national Prototype Kilogram.

**Cell**

The elementary unit into which two- and three-dimensional sampling units can be divided.

**Remark:** The dominant dimension of a cell is its height, and this dimension sets a cell apart from a stratum.

**Certified Reference Material**

An analysis sample that is prepared, certified and distributed by an authoritative institution, in which one or more elements or properties are

known with accuracy by definition, and with a high degree of precision by replicate measurements in different laboratories under carefully controlled conditions.

**Coefficient of Variation**

A measure for random variations between series of measurements, and numerically equal to the standard deviation as a percentage of the mean value for the parameter.

**Comminution**

The process of crushing, grinding and pulverizing required to prepare a gross sample to analytical fineness.

**Completely Randomized Sampling**

A sampling regime that is based on randomly collecting a set of primary increments from a sampling unit.

Remark: Completely randomized sampling regimes are not very efficient for sampling of bulk solids

**Composition Variance**

A measure for random variations in composition between individual particles, and thus, for the composition heterogeneity.

Remark: The sum of this composition variance and the distribution variance constitutes the sampling variance.

**Confidence Limits (Intervals and Ranges)**

The limits that form the boundaries within which 95 %, 99 % or 99.9 % of all measurements fall.

Remark: In chemistry and physics 95 % confidence intervals and ranges are commonly used.

**Consignment**

A quantity of material contained in one or more trucks, rail cars, barges, a unit train or a cargo aboard a seagoing vessel.

**Control Chart**

A graph that displays accuracy and precision characteristics of measurements.

Remark: Different types of charts such as charts for accuracy, charts for precision, and cumulative sum charts are used.

### **Control Limit**

A 95 % confidence range for a parameter on a control chart.

Remark: Upper and lower control limits are used in control charts for accuracy and precision

### **Correlation Coefficient**

A measure for interdependence between paired measurements, that may vary between  $-1$  and  $+1$  with the intermediate value of zero indicating a complete absence of interdependence.

### **Degrees of Freedom**

The number of independent comparisons that can be made among the set of measurements.

Remark: For a series of  $n$  measurements this number is  $n-1$ , but for a matrix of measurements the number of degrees of freedom is more complex.

### **Difference**

A deviation from a certified value, or a target value, that has not been identified as a random variation, a systematic error or bias, or a combination of both.

Remark: Imprecise measurements may obscure a bias for a longer period of time than precise measurements.

### **Distribution Variance**

A measure for random variations in the distribution of particles of all sizes throughout a sampling unit, and thus, for the distribution heterogeneity.

Remark: The sum of this distribution variance and the composition variance constitutes the sampling variance.

### **Equiprobable Sampling Space**

A sample space in which each possible outcome has an identical, finite probability.

### **Error (absolute)**

A statistically significant difference between a measurement, or the mean of a series of measurements, and the most reliable estimate of the unknown true value.



**Error (relative)**

A statistically significant difference between a measurement, or the mean of a series of measurements, and the most reliable estimate for the unknown true value, divided by this estimate for the parameter.

**Estimate**

A measurement, with a weighing device or on a sample, that is reported as a substitute for the unknown true value of the measured parameter.

**Gross Sample**

A quantity of bulk solids that consists of the complete set of primary increments from a sampling unit, either in the condition as collected or comminuted and/or divided as required.

**Increment**

A quantity of bulk solids collected by a single operation of a sampling device.

Remark: The mass of a primary increment may range from 100 grams for a fine mineral concentrate to more than 1,000 kilograms for mechanically collected increments from a run-of-mine ore at a high capacity bulk handling terminal.

**Interpenetrating Samples**

A set of two or more samples collected by storing consecutive primary increments alternately into two or more sample containers.

**Lot**

A quantity of bulk solids to be represented by a separate gross sample.

Remark: The mass of a lot may range from less than a single metric ton for a precious metal concentrate up to 10,000 metric tons for iron ore and metallurgical coal.

**Null Hypothesis**

The hypothesis that the mean difference between identifiably different measurements is statistically equal to zero.

Remark: The null hypothesis requires an alternate hypothesis that is formulated in such a manner that these hypotheses are mutually exclusive.