BS ISO 13909-7:2016

BSI Standards Publication

Hard coal and coke — Mechanical sampling

Part 7: Methods for determining the precision of sampling, sample preparation and testing

Documents
Document Sharing Hub

Full document details: BS ISO 13909-7:2016

National foreword

This British Standard is the UK implementation of ISO 13909-7:2016. It supersedes BS ISO 13909-7:2001 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/16, Solid mineral fuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2016. Published by BSI Standards Limited 2016

ISBN 978 0 580 83180 5

ICS 73.040; 75.160.10

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 June 2016.

Amendments issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: BS ISO 13909-7:2016

INTERNATIONAL STANDARD

ISO 13909-7

Second edition
2016-07-01

Hard coal and coke — Mechanical sampling —

Part 7: Methods for determining the precision of sampling, sample preparation and testing

Houille et coke — Échantillonnage mécanique —

Partie 7: Méthodes pour la détermination de la fidélité de l'échantillonnage, de la préparation de l'échantillon et de l'essai

Reference number
ISO 13909-7:2016(E)

Documents
Document Sharing Hub

© ISO 2016

Full document details: BS ISO 13909-7:2016

COPYRIGHT PROTECTED DOCUMENT

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub

Full document details: BS ISO 13909-7:2016

Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 General **1**
5 Formulae relating to factors affecting precision **2**
5.1 General 2
5.2 Sampling 3
6 Estimation of primary increment variance **4**
6.1 Direct determination of individual primary increments 4
6.2 Determination using the estimate of precision 5
7 Methods for estimating precision **5**
7.1 General 5
7.2 Duplicate sampling with twice the number of increments 5
7.3 Duplicate sampling during routine sampling 8
7.4 Alternatives to duplicate sampling 9
7.5 Precision adjustment procedure 9
8 Calculation of precision **10**
8.1 Replicate sampling 10
8.2 Normal sampling scheme 11
9 Methods of checking sample preparation and testing errors **12**
9.1 General 12
9.2 Target value for variance of sample preparation and analysis 12
9.2.1 General 12
9.2.2 Off-line preparation 13
9.2.3 On-line preparation 13
9.3 Checking procedure as a whole 13
9.4 Checking stages separately 14
9.4.1 General 14
9.4.2 Procedure 1 15
9.4.3 Procedure 2 18
9.4.4 Interpretation of results 21
9.5 Procedure for obtaining two samples at each stage 22
9.5.1 With a riffle 22
9.5.2 With a mechanical sample divider 22
9.6 Example 22
Annex A (informative) **Variogram method for determining variance** **26**
Annex B (informative) **Grubbs' estimators method for determining sampling precision** **34**
Bibliography **43**

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

This second edition cancels and replaces the first edition (ISO 13909-7:2001), which has been technically revised.

ISO 13909 consists of the following parts, under the general title *Hard coal and coke — Mechanical sampling*:

— *Part 1: General introduction*

— *Part 2: Coal — Sampling from moving streams*

— *Part 3: Coal — Sampling from stationary lots*

— *Part 4: Coal — Preparation of test samples*

— *Part 5: Coke — Sampling from moving streams*

— *Part 6: Coke — Preparation of test samples*

— *Part 7: Methods for determining the precision of sampling, sample preparation and testing*

— *Part 8: Methods of testing for bias*

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

Introduction

Two different situations are considered when a measure of precision is required. In the first, an estimate is made of the precision that can be expected from an existing sampling scheme and, if this is different from that desired, adjustments are made to correct it. In the second, the precision that is achieved on a particular lot is estimated from the experimental results actually obtained using a specifically designed sampling scheme.

The formulae developed in this part of ISO 13909 are based on the assumption that the quality of the fuel varies in a random manner throughout the mass being sampled and that the observations will follow a normal distribution. Neither of these assumptions is strictly correct. Although the assumption that observations will follow a normal distribution is not strictly correct for some fuel parameters, this deviation from assumed conditions will not materially affect the validity of the formulae developed for precision checking since the statistics used are not very sensitive to non-normality. Strictly speaking, however, confidence limits will not always be symmetrically distributed about the mean. For most practical uses of precision, however, the errors are not significant.

NOTE In the text, the term “fuel” is used where both coal and coke would be applicable in the context and either “coal” or “coke” where that term is exclusively applicable.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

Documents
Document Sharing Hub

Full document details: BS ISO 13909-7:2016

INTERNATIONAL STANDARD ISO 13909-7:2016(E)

Hard coal and coke — Mechanical sampling —

Part 7: Methods for determining the precision of sampling, sample preparation and testing

1 Scope

In this part of ISO 13909, formulae are developed which link the variables that contribute to overall sampling precision. Methods are described for estimating overall precision and for deriving values for primary increment variance which can be used to modify the sampling scheme to change the precision. Methods for checking the variance of sample preparation and testing are also described.

2 Normative references

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

ISO 13909-1, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*

ISO 13909-2, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*

ISO 13909-3, *Hard coal and coke — Mechanical sampling — Part 3: Coal — Sampling from stationary lots*

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 13909-5, *Hard coal and coke — Mechanical sampling — Part 5: Coke — Sampling from moving streams*

ISO 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13909-1 apply.

4 General

When designing a sampling scheme in order to meet a required precision of results, formulae are necessary that link certain fuel and sampling characteristics to that precision. The main factors to be considered are the variability of primary increments, preparation and testing errors, the number of increments and samples taken to represent the lot, and the mass of the samples. These formulae are derived in Clause 5. Methods for estimating the parameters used in those formulae are given in Clause 6.

Once a sampling system has been designed and installed, the precision which is being achieved on a routine basis should be checked. An estimate of the precision can be obtained from the primary increment variance, V_I, the numbers of increments, n, and sub-lots, m, (see Clause 5) and the preparation and testing variance, V_{PT}. The preparation component of V_{PT} is made up of on-line sample processing and off-line sample preparation.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

Sampling variance is a function of product variability, so the same number of increments, sub-lots, and preparation and testing errors will yield different precision with fuels that exhibit different product variability.

Depending on the extent to which serial correlation exists and which method of estimating primary increment variance is used, such an estimate could represent a considerable overestimate of the numerical value of the precision (i.e. indicate that it is worse than is really the case). In addition, in order for the results to be meaningful, large numbers of increments (in duplicate) would need to be prepared and analysed for the estimation of V_I and V_{PT}.

Quality variations obtained in the form of primary increment variances on existing systems are not absolute and therefore designers should exercise caution when using such results in a different situation. The estimated value of the primary increment variance, V_I, should be derived experimentally for each fuel and at each sampling location.

Whenever a sampling scheme is used for determining increment variance, the operating conditions should be as similar as possible to the conditions known, or anticipated, to prevail during the sampling for which the increment variance is needed, whether it be carried out by the same or by a different sampling system.

An estimate of the precision actually achieved can be obtained by taking the sample in a number of parts and comparing the results obtained from these parts. There are several methods of doing this, depending on

a) the purpose of the test, and

b) the practical limitations imposed by the available sampling procedures and equipment.

Where a sampling system is in existence, the purpose of the test is to check that the scheme is in fact achieving the desired precision (see Clause 7). If it is not, it may need to be modified and rechecked until it meets the precision required. In order to do this, a special check scheme should be devised which may be different from the regular scheme but which measures the precision of the regular scheme.

For regular sampling schemes, the most rigorous approach is that of duplicate sampling of sub-lots. In many existing mechanical sampling systems, however, the capacity of individual components and the interval between increments in the regular scheme is insufficient to allow the taking of extra increments. In such cases, duplicate samples can be constituted from the normal number of increments and the result adjusted for the smaller number of increments in each sample (see 7.3).

The need may arise to sample a particular lot and to know the precision of the result obtained (see Clause 8). Once again, a special check scheme needs to be devised, but in this case, it is the precision achieved by that scheme on that lot which is required. For the measurement of the precision achieved for a particular lot, replicate sampling is the best method.

Methods for detailed checking of preparation and testing errors are given in Clause 9. The results may also be used to provide data for the formulae used in Clause 5.

5 Formulae relating to factors affecting precision

5.1 General

Precision is a measure of the closeness of agreement between the results obtained by repeating a measurement procedure several times under specified conditions and is a characteristic of the method used. The smaller the random errors of a method, the more precise the method is. A commonly accepted index of precision is two times the sample estimate of the population standard deviation and this index of precision is used throughout this part of ISO 13909.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

If a large number of replicate samples, *j*, are taken from a sub-lot of fuel and are prepared and analysed separately, the estimated precision, *P*, of a single observation is given by Formula (1):

$$P = 2s = 2\sqrt{V_{\mathrm{SPT}}} \tag{1}$$

where

s is the sample estimate of the population standard deviation;

V_{SPT} is the total variance.

The total variance, V_{SPT}, in Formula (1) is a function of the primary increment variance, the number of increments, and the errors associated with sample preparation and testing.

NOTE The components of primary increment variance are the variance of sample extraction and the variance contributed by product variability. The variance contributed by product variability is generally, but not always, the largest source of variance in sampling.

For a single sample, this relationship is expressed by Formula (2):

$$V_{\mathrm{SPT}} = \frac{V_{\mathrm{I}}}{n} + V_{\mathrm{PT}} \tag{2}$$

where

V_{I} is the primary increment variance;

V_{PT} is the preparation and testing variance;

n is the number of primary increments in the sample.

5.2 Sampling

Where the test result is the arithmetic mean of a number of samples, resulting from dividing the lot into a series of sub-lots and taking a sample from each, Formula (2) becomes:

$$V_{\mathrm{SPT}} = \frac{V_{\mathrm{I}}}{mn} + \frac{V_{\mathrm{PT}}}{m} \tag{3}$$

where

m is the number of sample results used to obtain the mean.

Since a sample is equivalent to one member of a set of replicate samples, by combining Formulae (1) and (3), it can be shown that:

$$P = 2\sqrt{\frac{V_{\mathrm{I}}}{mn} + \frac{V_{\mathrm{PT}}}{m}} \tag{4}$$

Formula (4) gives an estimate of the precision that can be expected to be achieved when a given sampling scheme is used for testing a given fuel, the variability of which is known or can somehow be estimated. In addition, Formula (4) enables the designer of the sampling scheme to determine, for the desired precision and with fuel of known or estimated variability, the combination of numbers of increments and samples, respectively, which will be most favourable considering the relative merits of the sampling equipment and the laboratory facilities in question. For the latter purpose, however,

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

it is more convenient to use either or both Formulae (5) and (6), both of which have been derived by rearranging Formula (4).

$$n = \frac{4V_{\mathrm{I}}}{mP^2 - 4V_{\mathrm{PT}}} \tag{5}$$

$$m = \frac{4\left(V_{\mathrm{I}} + nV_{\mathrm{PT}}\right)}{nP^2} \tag{6}$$

NOTE Results obtained from solid mineral fuels flowing in a stream will frequently display serial correlation, i.e. immediately adjoining fuel tends to be of similar composition, while fuel further apart tends to be of dissimilar composition. When this is so, the estimates of precision of the result of a single sample based on primary increment variance and the variance of preparation and testing would indicate precision that is worse, i.e. numerically higher, than the precision actually achieved. The effect of serial correlation can be taken into account using the variographic method of determining variance given in informative Annex A.

6 Estimation of primary increment variance

6.1 Direct determination of individual primary increments

The direct estimation of primary increment variance can be accomplished with a duplicate sampling scheme comprised of several hierarchical levels which allows both the overall variance and the variance of preparation and testing to be estimated. The estimated variance of primary increments can then be obtained by subtraction of the variance of preparation and testing from the estimated overall variance. A number of primary increments is taken systematically and either divided into two parts or prepared so that duplicate samples are taken at the first division stage. Each part is prepared and tested for the quality characteristic of interest, using the same methods that are expected to be used in routine operations. The mean of the two results and the difference between the two results are calculated for each pair.

It is recommended that at least 30 increments be taken, spread if possible over an entire lot or even over several lots of the same type of fuel.

The procedure is as follows.

a) Calculate the preparation and testing variance, V_{PT}.

$$V_{\mathrm{PT}} = \frac{\sum d^2}{2n_{\mathrm{p}}} \tag{7}$$

where

d is the difference between pair members;

n_{p} is the number of pairs.

b) Calculate the primary increment variance, V_{I}.

$$V_{\mathrm{I}} = \frac{\sum x^2 - \frac{1}{n_{\mathrm{p}}}\left(\sum x\right)^2}{\left(n_{\mathrm{p}} - 1\right)} - \frac{V_{\mathrm{PT}}}{2} \tag{8}$$

where

x is the mean of the two measurements for each increment.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

An alternative method for estimating primary increment variance, V_I, is as follows:

$$V_I = \frac{\sum D^2}{2h} - \frac{V_{PT}}{2} \tag{9}$$

where

D is the difference between the means of successive pairs;

h is the number of successive pairs.

This method avoids the overestimation of sampling variance when there is serial correlation (see Note in 5.2) but can only be used if the primary-increment sampling interval at which the increments are taken is more than or approximately equal to the primary-increment sampling interval used when the scheme is implemented in routine sampling operations.

The most rigorous treatment of serial correlation is to use the variographic method given in Annex A. This takes into account both serial correlation and sampling interval effects, thereby avoiding overestimation of sampling variance and number of primary increments due to these factors.

6.2 Determination using the estimate of precision

The primary increment variance can be calculated from the estimate of precision obtained either using the method of duplicate sampling given in 7.2 or replicate sampling given in Clause 8 according to Formula (10) which is derived by rearranging the terms of Formula (4).

$$V_I = \frac{mnP^2}{4} - nV_{PT} \tag{10}$$

This value can then be used to adjust the sampling scheme if necessary.

7 Methods for estimating precision

7.1 General

For all the methods given in this Clause, the following symbols and definitions apply:

— n_0 is the number of increments in a sub-lot for the regular scheme;

— m_0 is the number of sub-lots in a lot for the regular scheme;

— P_0 is the desired precision for the regular scheme;

— P_W is the worst (highest absolute value) precision to be permitted.

In all cases, the same methods of sample preparation shall be used as for the regular scheme.

7.2 Duplicate sampling with twice the number of increments

Twice the normal number of increments ($2n_0$) are taken from each sub-lot and combined as duplicate samples (see Figure 1), each containing n_0 increments. This process is repeated, if necessary, over several lots of the same fuel, until at least 10 pairs of duplicate samples have been taken.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

A parameter of the fuel is chosen to be analysed, e.g. ash content (dry basis) for coal, or Micum 40 index for coke. The standard deviation within duplicate samples for the test parameter is then calculated using Formula (11):

$$s = \sqrt{\frac{\sum d^2}{2n_p}} \tag{11}$$

where

d is the difference between duplicates;

n_p is the number of pairs of duplicates being examined.

An example of results for coal ash is given in Table 1.

Table 1 — Results of duplicate sampling, % ash, dry basis

Sample pair no.	Duplicate values %		Difference between duplicates	
	A	B	$\lvert A - B \rvert = d$	d^2
1	11,1	10,5	0,6	0,36
2	12,4	11,9	0,5	0,25
3	12,2	12,5	0,3	0,09
4	10,6	10,3	0,3	0,09
5	11,6	12,5	0,9	0,81
6	11,8	12,0	0,2	0,04
7	11,8	12,2	0,4	0,16
8	10,8	10,0	0,8	0,64
9	7,9	8,2	0,3	0,09
10	10,8	10,3	0,5	0,25
			Total	2,78

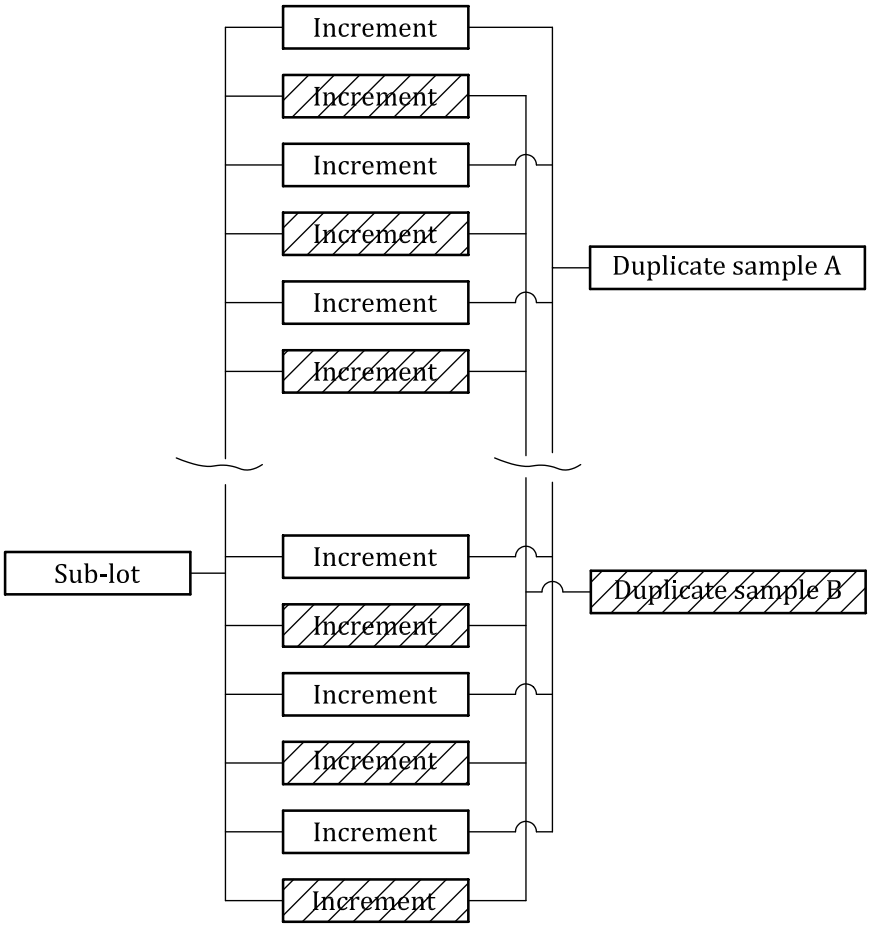
The number of pairs, n_p is 10. The variance of the ash content is therefore

$$s^2 = \frac{\sum d^2}{2n_p} = \frac{2,78}{20} = 0,139$$

and the standard deviation is:

$$s = \sqrt{0,139\,0} = 0,373$$

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016



Key

increment from regular scheme

extra increment for precision check scheme

Figure 1 — Example of a plan of duplicate sampling

The precision of the result for a single sub-lot is therefore:

$$P = 2s$$
$$= 2(0,373) = 0,75\ \%\,\text{ash}$$

The precision achieved for the mean ash of a normal lot sampled as m sub-lots is given by $2s/\sqrt{m}$. For example, if m = 10, then:

$$P = \frac{2(0,373)}{\sqrt{10}} = 0,235\ 9\ \%$$

These values of P have been calculated using point estimates for the standard deviation and represent the best estimate for precision.

If an interval estimate for the standard deviation is used, then on a 95 % confidence level, the precision is within an interval with upper and lower limits. These limits are calculated from the point estimate of precision and factors which depend on the degrees of freedom (f) used in calculating the standard deviation (see Table 2).

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

Table 2 — Factors used for calculation of precision intervals

f (number of observations)	5	6	7	8	9	10	15	20	25	50
Lower limit	0,62	0,64	0,66	0,68	0,69	0,70	0,74	0,77	0,78	0,84
Upper limit	2,45	2,20	2,04	1,92	1,83	1,75	1,55	1,44	1,38	1,24

NOTE The factors in Table 2 are derived from the estimate of s^2 obtained from the squared differences of n pairs of observations. Since there is no constraint in this case, the estimate as well as d^2 will have n degrees of freedom. The values in Table 2 are derived from the relationship:

$$\frac{ns^2}{\chi^2_{n,0,025}} < s^2 < \frac{ns^2}{\chi^2_{n,0,0975}}$$

The body of Table 2 gives the values for $\sqrt{n / \chi^2}$, which are multiplied by s to obtain the confidence limits.

For example, for the lot with 10 sub-lots used in the example above:

Upper limit = 1,75 (0,235 9) = 0,41 %

Lower limit = 0,70 (0,235 9) = 0,17 %

where the factors are obtained from Table 2 using $f = n_P$, i.e. 10. The true precision lies between 0,17 % and 0,41 % ash at the 95 % confidence level.

7.3 Duplicate sampling during routine sampling

If operational conditions do not allow the taking of $2n_0$ increments from each normal sub-lot or precision is to be determined during normal sampling, then, provided that all increments can be kept separate, adopt the following procedure for estimating precision.

Take the normal number of increments, n_0, from each sub-lot and combine them as duplicate samples each comprising $n_0/2$ increments (see Figure 2). Repeat this process, if necessary, over several lots of the same fuel until at least 10 pairs of duplicate samples have been obtained. In this case, the precision obtained using the procedure in 7.2 will be for $n_0/2$ increments. This estimate of precision is divided by the square root of 2 to obtain the estimate of precision for sub-lot samples comprising n_0 increments.

© ISO 2016 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016

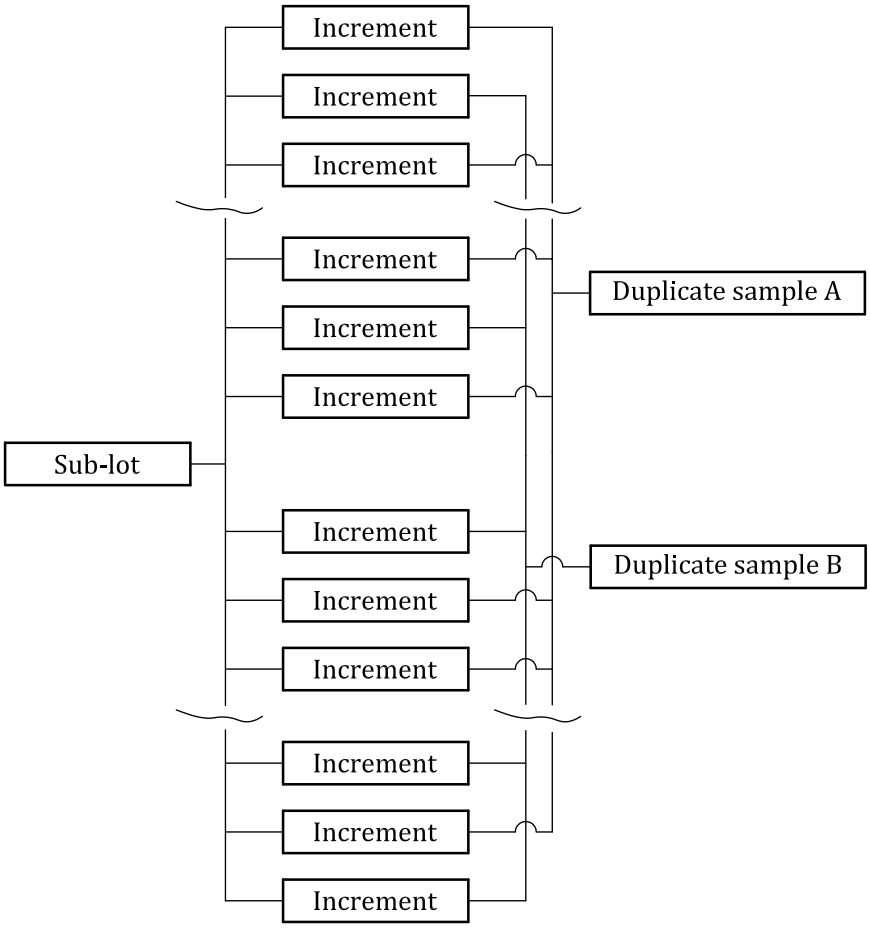


Figure 2 — Example of a plan of duplicate sampling where no additional increments are taken

7.4 Alternatives to duplicate sampling

At some locations, operational conditions of a sampling system do not allow duplicate samples to be collected with the assurance that no cross-contamination of sample material from adjacent primary increments occurs. In such cases, other methods have been found useful. An example of such a method, using Grubbs estimators, is given for information in Annex B.

This method involves collecting three samples from each of a minimum of 30 sub-lots of fuel. One sample is collected using the normal sampling scheme and two mutually independent systematic samples are collected by stopping a main fuel handling belt for collection of stopped-belt increments at preselected intervals.

7.5 Precision adjustment procedure

If the desired level of precision, P_0, for the lot lies within the confidence limits, then there is no evidence that this precision is not being achieved. However, if the confidence interval is wide enough to include both P_0 and P_W, the test is inconclusive and further data shall be obtained. The results shall be combined with the original data and the calculation done on the total number of duplicate samples.

NOTE The expected effect is reduction of the width of the confidence limits since the value of f in Table 2 will be greater.

© ISO 2016 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 13909-7:2016